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NONDESTRUCTIVE EVALUATION OF ADHESIVE BOND QUALITY

STATE-OF-THE-ART REVIEW

SwRI Project 17-7958-838

by

G. M. Light
Hegeon Kwun
SOUTHWEST RESEARCH INSTITUTE
San Antonio, Texas

June 1989

NONDESTRUCTIVE TESTING INFORMATION ANALYSIS CENTER

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This state-of-the-art report describes the bonding process, the destructive methods used to measure bond strength, and the various NDE methods that have been evaluated for determining the quality of a bond. These NDE methods include sonics, ultrasonics, acoustic emission, nuclear magnetic resonance, x-ray and neutron radiography, optical holography, and thermography. Each of these methods has shown some limited success in detecting debond conditions. At the present time, however, it appears that only the sonic, ultrasonic, and nuclear magnetic resonance methods have the potential capability to differentiate qualitatively the gradations between a good bond and a debond and thus provide a correlation to bond strength.

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ABSTRACT

Adhesively bonded materials are increasingly being used by industries to obtain stronger, lighter weight, and more durable structures. Many of these structures undergo tremendous environmental weathering and service stresses; and, consequently, the possibility of bonding failure is a major industrial concern, especially where rupture could cause severe harm to human operators. The nondestructive evaluation (NDE) community has been studying various methods for inspecting bonds before and during service; but, to date, bond strength cannot be directly measured non-destructively. The NDE approach has been used to obtain NDE test parameters (such as ultrasonic attenuation, velocity change, thermal dissipation, and mass absorption) from test samples and then to correlate the parameters with the actual bond strengths measured when these same samples were destructively assayed. The correlation is considered a qualitative bond evaluation; presently, only a destructive assay can supply a quantitative measurement for the strength of a specific bond.

This state-of-the-art report describes the bonding process, the destructive methods used to measure bond strength, and the various NDE methods that have been evaluated for determining the quality of a bond. These NDE methods include sonics, ultrasonics, acoustic emission, nuclear magnetic resonance, x-ray and neutron radiography, optical holography, and thermography. Each of these methods has shown some limited success in detecting debond conditions. At the present time, however, it appears that only the sonic, ultrasonic, and nuclear magnetic resonance methods have the potential capability to differentiate qualitatively the gradations between a good bond and a debond and thus provide a correlation to bond strength.

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I. CHARACTERISTICS OF ADHESIVE BONDING

A. Applications

1. Advantages and Disadvantages

Adhesive bonding is the process of joining materials by surface attachment with the aid of an adhesive. Because of its strength and reliability, adhesive bonding offers many important advantages over other joining methods such as welding, riveting, and mechanical fastening (1-4). Some advantages are that it

- Distributes stress more uniformly and minimizes areas of high stress concentration and, therefore, permits fabrication of lighter and stronger structures.
- Provides joints with smooth contours, allowing fabrication of more aerodynamically favorable structures.
- Allows joining of dissimilar materials such as metals to composites, rubbers to metals, or metals to metals. In the latter case, adhesive bonding minimizes the possibility of electrolytic corrosion problems.
- Permits easier fabrication of complex contoured parts.
- Provides good damping of sound and vibrations in a structure.
- Can be used as a seal against liquids or gases as well as an electrical insulator or conductor.
- May reduce the fabrication cost of a structure.

On the other hand, adhesive bonding has several disadvantages and limitations (1-4). Some of these are as follows:

- Does not permit visual examination of the bonded area; and, as a result, quality control and quality assurance are more difficult.
- Generally restricts upper service temperature to about 180 degrees C, although some special adhesives such as polyquinoxalines are available for limited use to about 370 degrees C.
- Requires tight control for processes such as surface preparation, material handling, and curing.
- Degrades under environmental exposure.
- May require long cure times, particularly where high curing temperatures are not used.

2. Typical Fabrication Applications

Adhesive bonding has been extensively used for fabrication of load-bearing structural components used in aircraft, helicopters, rockets, missiles, satellites, and space shuttles (1, 2, 5-7). The primary reasons are savings in weight and smooth joint contours. Specific examples of structures constructed by using adhesive bonding are airframes, wings, fuselages, rudders, doors, tail sections, and floor panels of aircraft (including helicopters) and bulkheads, nose-cones, nozzles, and motor casings of rockets or missiles.

Additionally, adhesive bonding has been widely used in other industries such as automotive, electronic, and building construction (1, 8-10). Examples of applications are fabrication of brakes and bodyshells of automobiles; fabrication of electrical equipment such as motors, transformers, generators, and microwave devices; and construction of tile floors, roofs, and mobile homes.

3. *Types of Adhesive Bonding Structures*

Types of adhesive bonding structures used in the aircraft and aerospace industries include (1, 4, 11-14):

- Metal-to-metal joints
- Metal-to-composite joints
- Metal-to-rubber joints
- Composite-to-composite joints
- Honeycomb sandwich structures composed of various combinations of honeycomb core materials (metal, paper, plastic) and facing materials (metal, composite, paper)
- Fiber-reinforced composite laminate structures.

Many joint designs or configurations (including lap, strap, stiffening, cylindrical, angle, corner, and flange) have been used in fabrication of adhesively bonded components. Examples of several joint designs are illustrated in Figure 1-1. A honeycomb sandwich structure is a laminar construction consisting of thin facings bonded to a relatively thick lightweight core as depicted in Figure 1-2. Fiber-reinforced composite laminate structure is made by bonding and curing two or more layers of material (called prepreg) together; the prepreg is produced by embedding layers of high-strength, small-diameter (about 10 microns) reinforcing fibers in a polymeric matrix (Figure 1-3). The layers of fibers are stacked in various sequences of orientation to produce desired mechanical properties. Stacking sequences are unidirectional or multidirectional, such as 0 -90 -0, 0 -45 -0, -45 45 45 -45, and 0 -45 -90 -45 -0.

Typical metals used for adhesive bonding structures in aircraft and aerospace industries include aluminum, magnesium, steel, titanium, stainless steel, and their respective alloys. Metal honeycomb cores are mostly made of aluminum. Fiber materials used for reinforcing composite laminates include carbon/graphite, glass, boron, aramid, and polyethylene.

B. Bonding Process

1. *Adhesive Types*

Adhesives used for structural bonding can be typically classified as thermosetting, thermoplastic, and elastomeric (1-4, 15). Many of the modern structural adhesives are, however, hybrids of these three (typically combinations of thermosetting and thermoplastic or elastomeric) to optimize strength, toughness, and high-temperature properties.

Thermosetting adhesives are synthetic, organic substances, which can be converted by chemical reaction into a permanently hard and practically infusible solid. They are activated by application of heat, catalysts, or a combination of both and are set permanently by cross-linking reactions. They have a higher modulus of elasticity; greater resistance to heat, chemicals, and adverse environments; but lower toughness than thermoplastics or elastomerics. The two most important thermosetting adhesives are those based on epoxy and phenolic.

Thermoplastic adhesives are synthetic, organic substances, which are hard when cold and softer when heated. Common thermoplastic resins include polyvinyls, polyacrylics, and polyethylene. Often employed in metal and plastic bonding, they are not usually suitable for structural applications--especially if subjected to elevated temperatures. Certain high-temperature thermoplastic adhesives such as polyamides, polyimides, and PEEK (polyetheretherketone) have high strength and good toughness and have been used in fabrication of advanced composite laminates (16-18).

Elastomeric adhesives are those organic substances that can be stretched to at least twice their original length without inherent loss of elastic properties. Natural and synthetic rubbers such as silicone, nitriles, polyurethane, and polysulfides belong to this category. They are often used as sealants, but lack the strength to be used alone for structural applications. They are widely used as a modifying agent for thermosetting or thermoplastic adhesives to improve toughness and peel strength of the system.

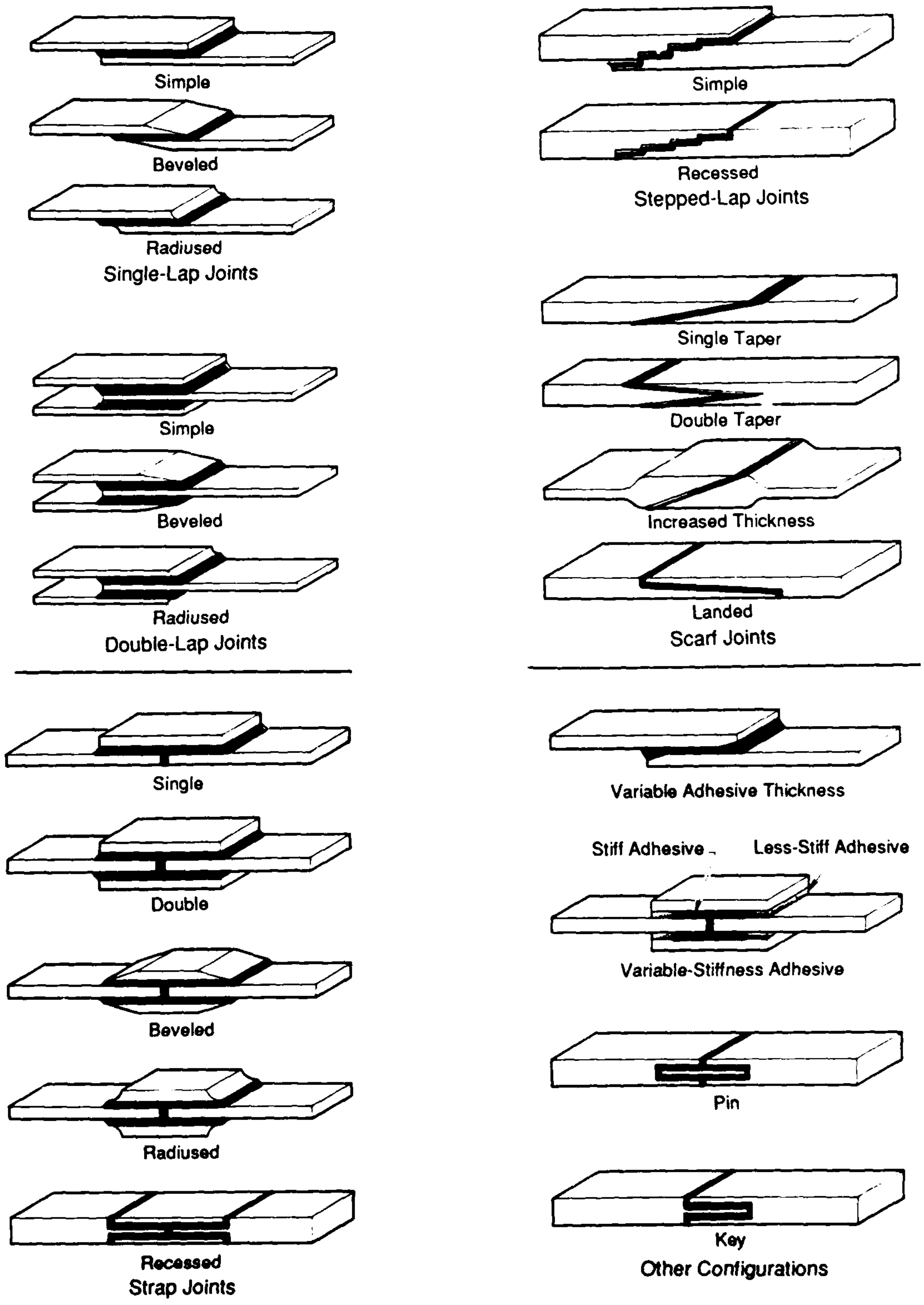

Figure 1-1. Examples of several joint designs [Ref. U.S. Army *DARCOM Handbook*, reproduced by permission of the U.S. Department of Defense]

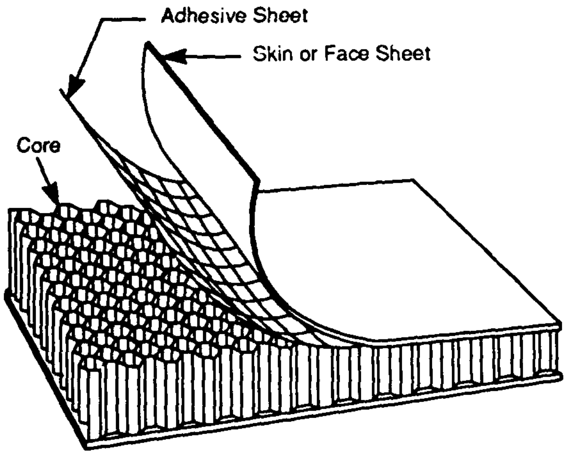

Figure 1-2. Honeycomb sandwich structure

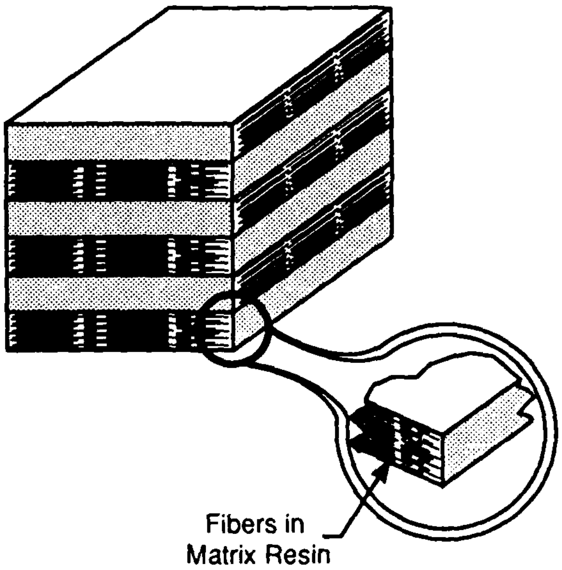

Figure 1-3. Schematic of a fiber-reinforced composite laminate structure (multidirectional)

Besides the basic resin, an adhesive consists of several other substances such as solvents, fillers, catalysts, diluents, hardeners, antioxidants, or placitizers. These are employed to control viscosity, reinforcement, shrinkage, temperature operating ranges, or thermal expansion coefficient; to activate, promote, and speed up the curing reactions; and to reduce material cost.

Adhesives are available in liquid, paste, film/tape, or powder form. Liquid adhesives are applied by brush, spray, roller, or mechanical spreaders. Paste adhesives are applied by spreading with mechanical tools such as a roller or trowel. Since the pastes have high viscosities, they can be used on vertical surfaces with little danger of drip. Film/tape adhesives are the most common in structural bonding applications. They offer a uniform bondline thickness and are convenient for application to flat or slightly curved surfaces. Film/tape adhesives are available in a pure sheet of adhesive or with cloth or paper reinforcement. These film/tape adhesives are also called "prepregs" and are generally stored under refrigeration. Film-type adhesives have a limited storage life compared with other forms. Powder adhesives, applied by dusting or spraying, are activated by applying heat.

2. *Sequential Steps*

A typical adhesive bonding process involves preparation of constituent materials (such as adherends, prepregs, adhesives, and/or primers), application of adhesives, assembly/lay-up, curing, and final finishing as illustrated in Figure 1-4 (1, 4, 19). To reduce fabrication time, improve reliability, and maintain a consistent quality, many steps of the bonding process have been automated; and the trend will continue (19-21). Each bonding process, shown in the figure, is briefly described in the following five subsections, respectively.

a. *Material Preparation*

Adherends. Of the processes used in preparing materials for bonding, surface treatment of adherends plays the most important role in determining the quality and reliability of the bonded structure. Surface preparation varies depending on the specific substrates employed, the design of parts, and the adhesive or primer used. In general, the process involves the following steps: (1) cleaning, (2) rinsing, (3) chemically treating, (4) rinsing, and (5) drying. Before surface

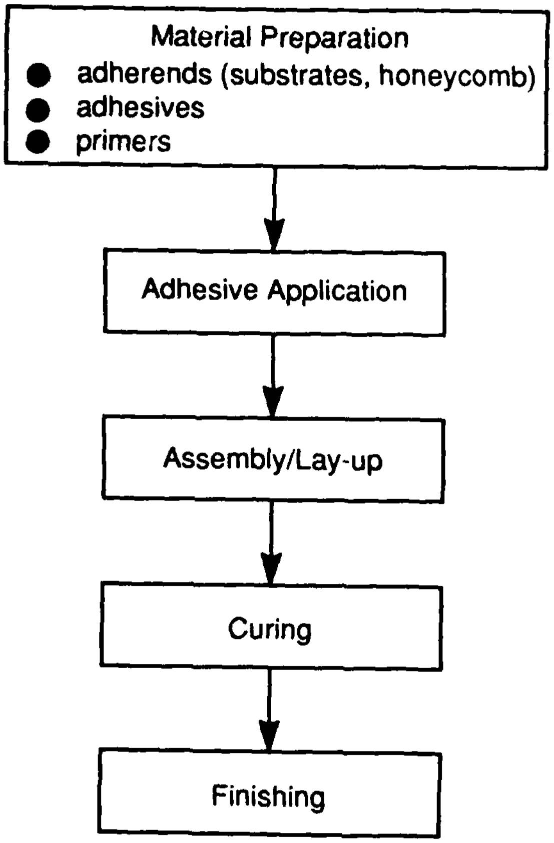

Figure 1-4. Typical adhesive bonding process

preparation, parts are prefitted to ensure that faying surfaces have the proper clearance.

For cleaning the surface, various methods are used including solvent, alkaline, ultrasonic, and vapor degreasing; abrading (by sanding or filing); and sandblasting (1, 4). When any chemicals are used in the cleaning operation, the parts are rinsed thoroughly in distilled or deionized water. When abrading or sandblasting is used, the dust particles created are removed by vacuuming or blasting with clean filtered air.

Chemical treatment is employed for metal or plastic substrates, excluding honeycomb cores, to make their faying surface receptive to adhesion. A wide variety of chemical treatment methods has been used depending on the substrate material (1, 4). For example, chromic-sulfuric acid [etch known as FPL (Forest Products Laboratory) etch] and phosphoric, chromic, and sulfuric acid anodizing have been used for aluminum (1, 4, 5, 22); nitric-hydrofluoric acid pickling, chromic acid anodizing, and alkaline peroxide etch for titanium (1, 4, 23-25); and hydrochloric acid and nitric-phosphoric etch for steel (1, 4, 26).

The chemical treatment is followed by rinsing in distilled or deionized water and then drying in clean air. Each step of the surface-preparation process is performed at a specified temperature (typically room temperature to 100 degrees C) and for a specified amount of time (typically a few minutes to half an hour).

In many cases, the faying surface of adherends is further treated by priming or conversion coating to enhance bonding and retard environmental degradation such as corrosion or water intrusion. Priming typically involves spraying a thin coating of primer with a thickness of a few to several microns to the faying surfaces and drying and curing the primer at a specified temperature for a predetermined amount of time. Various liquid or paste adhesives such as epoxy, phenolic, and silicone or coupling agents such as silane are used as primers.

Conversion coating consists of applying a chemical coating to a desired thickness (1) on the surface of metallic adherend after cleaning, rinsing in a chemical solution such as chromate, and drying (4, 26, 27). Conversion coatings are applied by dipping, spraying, or brushing. Such coatings are corrosion resistant and provide a good wetting surface for the adhesive.

For preparing metallic honeycomb cores, vapor degreasing, solvent cleaning, or light sanding followed by vacuuming or blasting with clean air are used (1). Plastic and paper cores are normally bondable as received. If contaminated, the core may be vapor greased. Light sanding followed by vacuuming or blasting with clean air may also be employed. If evidence of moisture is observed, the core is dried in an oven.

Adhesives. When film or tape adhesives are used, preparation involves typically (1) removing from a storage which is generally refrigerated, (2) thawing to room temperature, and (3) cutting to the required size and shape. For cutting, mechanical, laser, or water-jet cutters are used (7). When other adhesive forms such as liquid, paste, or powder are used, preparation involves weighing and mixing of base and additive materials such as catalysts, curing agents, hardeners, fillers, or placitizers. If the materials were stored cold, they must be warmed to room temperature before weighing and mixing.

Primers. Primers are prepared in a manner similar to the way liquid or paste adhesives are prepared.

b. Adhesive Application

Adhesives are applied to the substrates within typically a few hours after the surface preparation treatment. Various methods such as spray, brush, roller, and spreader are used for applying adhesives in liquid, paste, or powder form. Film or tape adhesives are applied by placing them on the mating surface of the substrate. Then wrinkles are worked out, all air-trapped air bubbles are removed, and the film is trimmed to the desired size. A tacking paste may be used to hold the film in place and to prevent slippage or movement during assembly.

c. Assembly/Lay-Up

After the adhesives are applied, the components are joined and assembled in a tool or holding fixture. For laminate structures, prepregs are laid up in a predetermined sequence of fiber alignment. Tools or holding fixtures hold the detail components in place, apply the necessary pressure, and sometimes transfer or apply the necessary heat for curing. Examples of assembly tools or fixtures are dead weight, vacuum bags, autoclave, clamps, and press (1). Provisions for the escape or breeding of gas released during curing are made. The assembly/lay-up is done in a well controlled atmosphere including typically 20- to 60-percent relative humidity, temperature between 18 to 32 degrees C, and clean air (4).

d. Curing

After the assembly, the adhesive bonds are cured according to a predetermined curing procedure specifying time-temperature-pressure requirements during the cure cycle. For elevated-temperature curing, the heat-up and cool-down rates and maximum temperature limits are also controlled. The gas released during curing is removed by vacuuming to eliminate debonds and porosities in bonding. During the cure cycle, pressure is maintained over the entire bonded area to obtain a uniformly thin adhesive bond layer and to overcome viscosity of the adhesive at the curing temperature, internal pressure exerted by the release of adhesive solvents or water vapors, and surface imperfections and dimensional mismatch between the mating surfaces (1).

e. Finishing

Finishing involves removing excess cured adhesives, trimming and machining the cured assembly to the desired dimensions, and sealing and painting the assembly.

3. Bonding Mechanisms

While many theories of adhesion have been proposed and debated, the detailed mechanisms involved in adhesive bonding or adhesion of two surfaces are not yet fully understood. This inability to measure the intrinsic adhesion forces acting across the adhesive/adherend interface has hindered the development of a comprehensive adhesion theory and the understanding of the detailed mechanism (28, 29). No single theory advanced so far can explain adhesion in a general all-encompassing way (4). In the following paragraphs, a brief description is given of the four main mechanistic theories proposed: mechanical interlocking, diffusion, electrostatic, and adsorption (4, 28-30).

Mechanical Interlocking Theory. In the mechanical interlocking theory, mechanical interlocking or keying of adhesive into the irregularities of the substrate surface provides the adhesion forces. For certain cases such as the adhesion of polymers to textiles and bonding of porous substrates, this mechanism

appears to be the major source of the intrinsic adhesion forces. However, it is believed that the frequently observed increase in measured joint strength with mechanical roughening is due to other mechanisms associated with a resulting change in physical and chemical properties of the surface with roughening.

Diffusion Theory. The diffusion theory is based on the mutual diffusion of polymer molecules across the interface between two polymers to cause adhesion. This mechanism requires that the adhesive and substrate polymers be mutually soluble and the macromolecules have sufficient mobility. Where the solubility of the material is not similar or one polymer is highly crosslinked and crystalline or is above its glass transition temperature, interdiffusion is an unlikely mechanism.

Electrostatic Theory. In the electrostatic theory, adhesion is caused by attractive electrostatic forces which arise from a double layer of electrical charge formed at the interface when two different electronic band structures of adhesive and substrate are in contact. The relevance and relative importance of this mechanism is still vague.

Adsorption Theory. Adsorption is the most generally accepted adhesion theory. Adhesion comes from the attractive surface forces acting between the atoms in the two surfaces when the two are in intimate molecular contact. The most common forces are Van der Waals and hydrogen bond (these are referred to as secondary bonds); other forces are ionic, covalent, and metallic bonds (these are referred to as primary bonds).

Van der Waals forces are attributed to different effects: (1) dispersion (or London) forces arising from internal electron motions which are independent of dipole moments and (2) polar (or Keesom) forces arising from dipole-dipole interactions. The hydrogen bond is formed through the attraction between a hydrogen atom and an electronegative atom such as oxygen, nitrogen, or fluorine. Primary interfacial bonding may occur in certain circumstances and may be responsible for the enhanced joint strength and resistance of the interface to attack by moisture resulting from the use of primers, promoters, or coupling agents.

When comparing the general magnitudes of these different types of forces (31), the Van der Waals forces are the weakest. (See Table 1-1.) At small intermolecular separations, however, dispersion forces are sufficiently strong to provide good bond strengths. For example, at molecular contact (separation of about 4 to 5 A), the attractive force between two perfectly plane parallel plates is about 10^9 to 10^{11} dynes/cm^2 (14 to 1400 Klbs/inch^2); at a separation of 10 A, the force is about 10^8 to 10^{10} dynes/cm^2 (1.4 to 140 Klbs/inch^2) (28). Although secondary bonding forces alone can provide good joint strengths at the interface, it is believed that the additional presence of primary bonding forces is crucial for establishing environmentally stable interfaces (32).

Table 1-1

GENERAL MAGNITUDE OF DIFFERENT TYPES OF INTERMOLECULAR FORCES
[Ref. (31), reproduced by permission of publisher Marcel Dekker, Inc.]

Type of Force	Energy (kcal/mole)
Ionic	140 - 250
Covalent	15 - 170
Metallic	27 - 83
Hydrogen bonds	Up to 12
Dispersion	Up to 10
Dipole-induced-dipole	Up to 0.5

C. Quality and Strength

Quality of a bond is related to the functional usefulness of the bonded assembly. Although no definition is universally accepted, the bond quality generally refers to absence of defects such as debonds, delaminations, voids, or foreign substances; mechanical strengths of the bonded joint; and joint durability in the expected service environment. A better quality bond generally implies that the bonded joint has less amount of defects and a higher mechanical strength and a longer durability.

Bond strength generally refers to the unit load, which is applied in tension, compression, flexure, peel, impact, cleavage, or shear, required to break an adhesive assembly with failure occurring in or near the plane of the bond (1, 4). Therefore, bond strength is called by various names such as tensile, shear, flexural, peel, impact, and fatigue, depending on the load type. Standard test methods for measuring these strengths in the laboratory with the use of standard test samples are available. Table 1-2 lists some of the test standards adopted by the American Society for Testing and Materials (ASTM). These standard test methods are destructive and are not meant to be used for determining the bond strength of a joint assembly in service.

Strictly speaking, bond strength should refer to the interfacial adhesion force per unit area acting between the adhesive and the adherend. Current technology, however, is incapable of measuring such interfacial forces. It has been well known that the bond strengths measured per standard test methods do not correlate well with the interfacial adhesion energies theoretically calculated. This poor correlation is attributable to various factors influencing the mechanically measured joint strength including the sample geometry and loading conditions used, flaw location, and failure mode (failure may not occur at the adhesive-adherend interface). Lack of appropriate means for measuring interfacial forces is apparently the major reason for the general use of the mechanically determined joint strength as the bond strength. Currently, no consensus exists on defining quality in a quantitative way based on defect content and location, mechanical strength, and durability data.

Many factors influence the quality of bond (1, 2, 4, 33-37) including:

- Bonded joint type and geometry
- Type of adherend material
- Adhesive type and composition and type of additives
- Treatment of adherend surface and resulting physical and chemical properties of the surface such as roughness, cleanliness, and wettability (25, 29, 38-42)
- Primer type (43-46)
- Environmental condition such as humidity, temperature, and exposure time during the bonding process (19, 47, 48)
- Curing parameters such as temperature, pressure, heat-up rate, and curing time (49-51)
- Bondline thickness (52, 53)
- Residual stress in the adhesive (54)
- Presence of defects induced during fabrication or service
- Workmanship
- Environmental degradation due to, for example, moisture, corrosion, vibration, shock, impact, temperature cycling, fatigue loading, and bacterial degradation (55-60)

In reality, the only way to determine the true strength of a bond is to destructively test it. Since this is not acceptable, one option is to perform a statistical evaluation by destroying and quantifying some number of production bonds. But this, too, is not acceptable to the manufacturer because it is prohibitively expensive. The next best choice is to fabricate test samples at the same time and under the same conditions as the production bonds.

In practice, the fabrication goal is to know that the production bond is as good as the bond in the test samples that are destructively analyzed to verify the fabrication process. Generally, the production bonding process is assumed to be equivalent to the process used on the test sample. However, many parameters affecting the final strength of a bond cannot be tightly controlled, so this assumption may be inappropriate.

Table 1-2

LIST OF ASTM STANDARD TEST METHODS
FOR MEASURING STRENGTHS OF BONDED JOINTS

ASTM Standard	Title
D897-78	Standard Test Method for Tensile Properties of Adhesive Bonds
D903-49	Standard Test Method for Peel or Stripping Strength of Adhesive Bonds
D905-49	Standard Test Method for Strength Properties of Adhesive Bonds in Shear by Compression Loading
D950-82	Standard Test Method for Impact Strength of Adhesive Bonds
D1002-72	Standard Test Method for Strength Properties of Adhesives in Shear by Tension Loading
D1062-78	Standard Test Method for Cleavage Strength of Metal-to-Metal Adhesive Bonds
D1184-69	Standard Test Method for Flexural Strength of Adhesive Bonded Laminate Assemblies
D2919-84	Standard Test Method for Determining Durability of Adhesive Joints Stressed in Shear by Tension Loading
D3163-73	Standard Test Method for Determining the Strength of Adhesively Bonded Rigid Plastic Lap-Shear Joints in Shear by Tension Loading
D3164-73	Standard Test Method for Determining the Strength of Adhesively Bonded Plastic Lap-Shear Sandwich Joints in Shear by Tension Loading
D3166-73	Standard Test Method for Fatigue Properties of Adhesives in Shear by Tension Loading

Nondestructive testing can provide the opportunity to obtain useful information about the production bond in an acceptable, cost-effective means. While bond strength can only be absolutely measured by a destructive test, NDE methods can directly measure some valuable parameters indicative of the bond interface condition. This interface does greatly affect the ultimate strength of the bond. Thus, it is the goal of NDE to correlate the effect of bond interface conditions on the NDE data to the actual bond strength data obtained destructively so that NDE can provide some information about the quality of the bond (relative to the destructively assayed test samples). By properly correlating specific NDE parameters to the results obtained through the destructive assay, a bond quality gauge could be developed for each type of bond and geometry.

The premise that NDE is not used to directly measure bond strength, but can only be used to determine bond quality (relative to bond-strength data obtained destructively) is a key point for utilizing the information contained in this report.

D. Failure Mechanisms

Failure of adhesive joints usually results from the initiation and propagation of flaws such as cracks, debonds, and delaminations. Depending on the location of the fracture surface, the failure is generally divided into two modes, adhesive and cohesive (1, 4). Adhesive failure is fracture at the interface between the adhesive and adherend (Figure 1-5). Cohesive failure is fracture in the adhesive so that a layer of adhesive remains on both adherends (Figure 1-5). Generally, failure occurs in mixed modes and is expressed as a certain percentage cohesive or adhesive.

The failure mode of a bond is sometimes used as a criterion for quality of bonding; for example, a good bond would fail cohesively within the adhesive layer or one of the adherends, but not adhesively at the adhesive/adherend interface. However, because some bonds that fail adhesively can exhibit greater mechanical strength than a similar joint bonded with a weaker adhesive, which fails cohesively, determining quality based on the failure mode is not recommended (4).

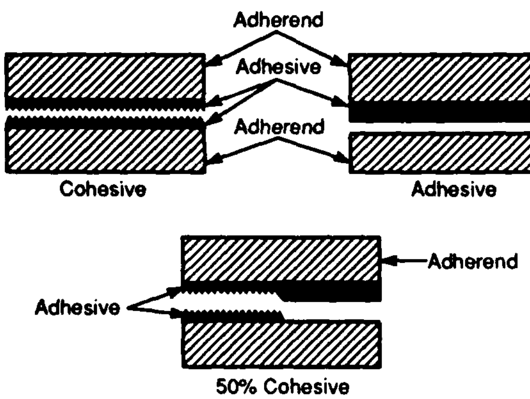

Figure 1-5. Cohesive and adhesive bond failure

Similarly with adhesion, the detailed mechanisms of joint failure are not well understood including the mechanisms of defect initiation and propagation toward the critical size. Defects such as debonds, cracks, and delaminations, however, can be initiated and propagated by fatigue; impact; static and dynamic loading; and weakening of adhesive, substrate, and the adhesive/adherend interface through aging and environmental degradation, or by a combination of these (2, 54, 57, 61-64). Microscopic defects preexisting in the material or induced during fabrication due to imperfect processing can also act as the initiators. The defect propagation and failure mode are dependent on various parameters including the material type, joint design, stacking sequence, environmental condition, type of surface treatment, and quality of bonding process.

Adhesives can be weakened through thermal degradation and breakdown, biological attack, or hydrolysis (2, 54, 62). Substrate weakening occurs through corrosion of the substrate surface or weakening of the oxide layer on the surface (2, 35, 36, 54, 62, 65). Matrix resin of fiber-reinforced composite materials can be weakened in humid or aqueous environment through corrosion, hydrolysis, depolymerization, or microcracking due to swelling caused by water uptake (66-70). The weakening of the interface between the adhesive and substrate occurs through desorption or displacement of the adhesive by the intrusion of water or other liquids (2, 54).

II. NDE METHODS AND TECHNIQUES

A. General Discussion

Over the last 20 years, a large amount of NDE research and development (R&D) has been conducted to determine how to detect and predict the quality of bond in adhesively bonded structures (71-131). These methods investigated include sonics, ultrasonics, holography, radiography, thermography, acoustic emission, and nuclear magnetic resonance. In this section, the state of the art for each of these methods will be discussed.

Some of these methods have had little success. In a 1974 general review of the NDE of adhesive bonds, Norriss (71) reported on several techniques including x-ray, holography, Vacuum cup (Avro bond tester), Resonance Fokker Tester, eddy sonic Shurtronic, ultrasonic Sondicator, heat-sensitive coatings and papers, acoustic impedance and emission, and through-transmission and pulse-echo UT. The most usable methods seemed to be the Fokker Bond tester, Sondicator, through-transmission UT, and acoustic impedance. However, most of these could only detect voids. The preferred method was Fokker Bond Tester for sheet-to-sheet joints; and for the core-to-sheet joints, radiography and through-transmission UT worked best.

In a more recent report (1984), Teagle (72) reported that no single NDE technique solved all problems. His studies found that radiography could be used to detect damage to honeycomb, presence of debris and contaminants, and lack of bond due to absence of adhesive. Conventional UT with 1 to 10 MHz can be used in the through-transmission mode to determine attenuation, which gave some information about debonds, delaminations, and porosity; but these defects cannot be easily distinguished from one another. Approximately the same information can be obtained with pulse-echo UT. Frequency analysis of reflected waves gives information about how the waves have interacted with the materials in the inspection path and can help discriminate between the different bonding problems.

Thermography can successfully monitor the variation in thermal conductivity due to defects in the bonded region. Thermographic techniques, such as infrared (IR) scanners and cameras, liquid crystals, treated paper, paints, and phosphors, and thermometric techniques, such as thermocouples and radiometers, were evaluated; but several problems were found. First, it is difficult to get suitable heat/cold sources; second, high anisotropic materials have different thermal properties in different directions, which cause data interpretation problems; and, third, metallic bonded structures can only hold temperature gradients for a short time.

Zurbrick (73) reported that the most critical area for NDE R&D was the substrate surface preparation prior to bonding. He developed an equation for predicting bond strength based upon surface energy in which all the controlling variables could potentially be measured using NDE. The equation is as follows:

$$E_{SL} = E_S - E_L \cos\theta \qquad (1)$$

where E_S is the solid-surface energy (in a vacuum), E_L is the liquid-surface free energy (in a vacuum), E_{SL} is the solid-liquid interface surface-free energy (assumed to be the bond adhesive strength), and θ is the contact angle at the three-phase point.

The bond strength is the energy stored in the adhesive volume at the moment of fracture. The bond strength is primarily determined by the thickness of the bondline, which greatly affects the energy stored in the bond. The ultimate bond strength is related to the solid-surface energy, liquid-surface energy, thickness of the bond, contact angle, and strain. Bond quality can be predicted if bond thickness, contact angle of adhesive-to-substrate, and substrate surface-free energy can be measured nondestructively.

To measure the variables just listed, NDE techniques including exo-electron emission, ultrasonic gas-phase transmission, electric

field reflectrometry, and light specular reflectance were used by Zurbrick. Substrate surface-free energy had the most dramatic linear effect. The contact angle was less dramatic. Effective strain was less than anticipated, and the others behaved in a similar manner. Zurbrick's basic premise was that the adhesive bonding is controlled by the surface electron-energy state. He took light reflections from the surface using a 45-degree incident white light and a detector at 45 degrees. His data indicated that the reflected light was inversely related to the calculated substrate surface-free energy.

Segal et al. (74) handled the problem somewhat differently. They divided the problem of nondestructively determining adhesive bond integrity into four parts: (1) detection of the unbond, (2) prediction of the cohesive quality of the bond, (3) detection of adhesive failure, and (4) prediction of a combined cohesive/adhesive quality.

Almost all NDE methods have been used to inspect for debonds; that is, UT, eddy sonic, radiography (x-ray, neutron, tracers), thermography (IR, moving heat source, liquid crystals, thermochromatic coating), optical holography, acoustic emission, liquid penetrants, and microwaves. The consensus of this work was that the most accurate method for detecting voids, porosity, and excess adhesive was radiography. Neutron radiography is good for bonded metallic structures; for structures containing composite, x-ray is more effective. However, access to both sides of the part is needed. When access to one side only is available, then optical holography or pulse-echo ultrasonics is most useful.

For detecting debonds, the most common NDE method is ultrasonics. This can be done by using tapping, pulse echo, through transmission, resonance, acoustic impedance, spectrum analysis, logarithmic decrement, acoustic holography, and critical angle reflectivity.

The following subsections discuss the work conducted using the various methods and specific techniques.

B. Sonic Techniques

Sonic techniques refer to inspection processes that utilize mechanical impulses to produce low-frequency vibrations. These vibrations are usually in the audio range, and the propagation of the frequencies is affected by the mechanical condition (e.g., flaws and density variation) of the part under inspection.

Several sonic inspection techniques have been used over the years for detecting the quality of bond of an adhesively bonded structure. These include the coin tap test, mechanical impedance, and mechanical resonance (75).

1. Coin Tap Test

The coin tap test has been used for many years as a quick yet sensitive technique to find large debond areas in laminated and honeycomb structures. This test is usually very subjective, but work by Adams and Cawley (76) has shown that the technique can be quantized. They point out that the difference between tapping a good or a defective region is due to the change in force input. The local structural stiffness is affected by the presence of the defect, and the force/time characteristics are dependent upon the local structural impedance. Adams and Cawley found that the impact on a good region had a higher amplitude and a shorter time duration (or contained higher frequencies), while the impact on a defective region had a lower amplitude and a longer duration (or contained lower frequencies), as illustrated in Figure 2-1. Therefore, by incorporating a force transducer in the impact hammer, the location of adhesive defects can be found. The sensitivity of the technique has not been evaluated.

2. Mechanical Impedance

The mechanical impedance technique works on the principle that the localized impedance of a sample is dependent upon the defect condition of the sample at that point (75). The point impedance of a

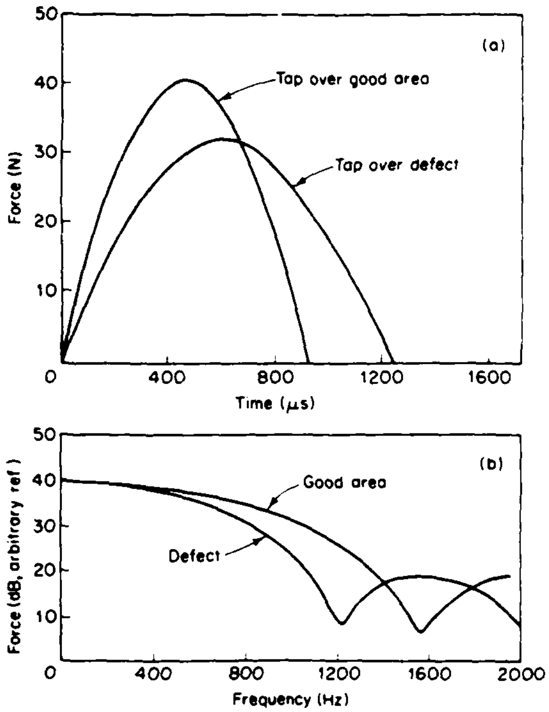

Figure 2-1. Force/time records for impacts on debonded and sound regions along with associated frequency responses [Ref. (76), reproduced by permission of Gordon and Breach, Science Publishers, Inc.]

structure can be determined by applying a force and measuring the resultant velocity of the structure due to the force. That is,

$$Z = F/v \qquad (2)$$

where Z is the local impedance, F is the harmonic force input to the structure, and v is the resultant velocity of the structure.

Most commercially available instruments work between 1 and 10 kHz. As the impedance of the structure decreases, the quality of the bond decreases. The impedance, however, is very dependent upon the flexibility of the base structure. If the structure becomes more flexible, the impedance of the defective region can be higher or lower than that of the good region, depending upon the input frequency; the test, therefore, is not very reliable. Because the sensitivity of this technique is directly related to the contact stiffness of the material being tested, the technique does not work well with a composite structure.

3. Mechanical Resonance

A planar debond was modeled as a plate restrained around the edges by the surrounding structure (76). As the frequency of excitation increased, the debond resonated. At resonance, the impedance over the defective region decreased, and the response for a given force input increased. If the frequency were close to the fundamental frequency, the response amplitude for the defective region would be much higher than the surrounding region. The layers above a defect were modeled as a disc, and the resonant frequency of the disc was directly related to the depth of the defect and the radius of the defective region, as well as the square roots of Young's modulus, the density, and a function of Poisson's ratio. The typical operating frequencies for instruments using this technology were 20 to 30 kHz.

Another mechanical resonance method used to evaluate adhesive bonds utilized a lower frequency. Low-frequency (~10 Hz) dynamic measurements of the shear modulus of epoxy phenolic and other adhesive systems were shown to vary greatly during curing and, in fact, to provide a measure of the degree to which curing had progressed (77). Damping of the vibrations of adhesive rod specimens also correlated with cure and strength. A plot of the log decrement of decay of free oscillations measured versus bond strength shows that the lower the adhesive bond, the higher the decrement. This is illustrated in Figure 2-2.

Meyer and Chapman (78) reported that the Ford Motor Company has used NDE to monitor the bond quality of adhesively joined fiberglass-reinforced plastic (FRP) since late 1970. Their technique used the low-frequency Automation Industries' Sondicator S-2B. They found that the three factors affecting the strength of FRP were the substrate, primer, and adhesive. To address the potential problem with quality assurance, Ford developed three concepts: quality bonding environment, bond-strength profile, and bond merit factor (BMF). The reason Ford

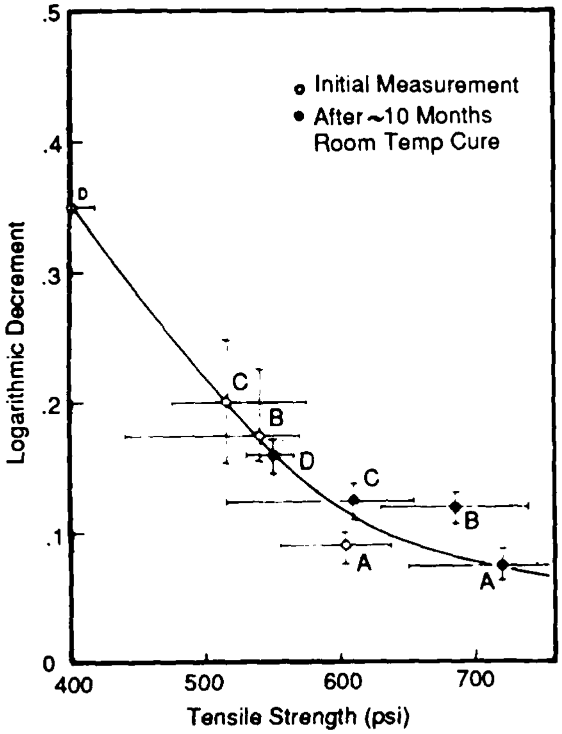

Figure 2-2. Logarithm decrement of HT-424 bonded panels versus tensile strength at 7.2 kHz [Ref. (77), reproduced by permission of the American Society for Nondestructive Testing (ASNT)]

came up with the bond merit factor was to answer the question: If a partial bond exists and is not detected, is it serious to the product? The equation for the BMF is given by

$$BMF = (L - C - P/2)/L \quad (3)$$

where L is the length of the bond inspected, C is the length of completely debonded joints, and P is the length of any joint found partially bonded.

In a review paper, Thompson et al. (77) pointed out that a number of survey experiments have suggested that the adhesive bond quality of honeycomb sandwich panels also can be nondestructively determined by measurements of vibrational response. The data showed that both the damping characteristics of panel vibrations as a whole and the velocity of the propagation of elastic waves that travel along the surface and the sample bondline can be correlated with destructively determined bond strengths. These results are a consequence of the viscoelastic properties of the adhesive whereby changes in the elastic moduli and damping capacity of the adhesive can be related to the molecular structure and hence the quality of the adhesive.

C. Ultrasonic Techniques

Ultrasonic techniques refer to inspection processes that utilize a piezoelectric-element transducer to generate ultrasonic energy into a part. This energy interacts with the part under inspection and is propagated back to either the transmitting transducer or a separate receiving transducer. The ultrasonic energy can be transmitted in various modes (e.g., longitudinal, shear, or surface waves) and at various angles. The condition of the material under inspection affects the amplitude and frequency of the ultrasonic waves as they propagate through the material. In some inspections, only the amplitude information is used to evaluate bond quality; and in others, only the frequency information is analyzed.

A tremendous amount of work has been conducted to evaluate the capability of ultrasonic techniques to determine the quality of adhesive bonds. The work has consisted of studying longitudinal, shear, surface, and interface waves. Techniques include those that measure the response of the signal reflected from or transmitted through the bonded interface region and those that interact directly with the adhesive layer. These techniques are described in more detail in the following paragraphs.

1. Amplitude-Domain Reflection/Transmission at the Bond Interface

The amplitude-domain reflection/transmission techniques attempt to determine the quality of the bond by evaluating the interaction of the ultrasonic beam with the adhesive boundary interfaces. For the pulse-echo mode, as the ultrasonic beam interacts with the boundary of the adhesive, certain reflections should occur. [See Figure 2-3, Ref. (79).] If a good bond exists, little reflection should be observed (except due to pure impedance matching). If a defective bond

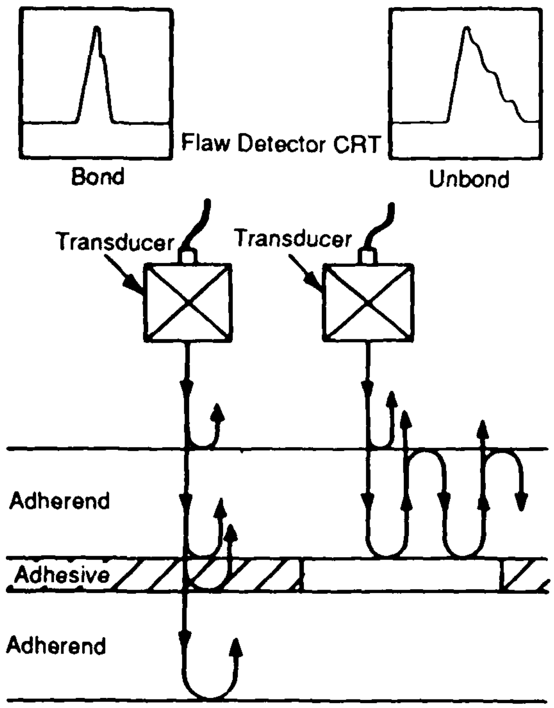

Figure 2-3. Pulse-echo techniques [Ref. (79), reproduced by permission of the Academic Press]

exists, then reflection would increase because the impedance mismatch is due not only to the impedances of the adherents and adhesive, but also to the presence of air or other inclusions. The through-transmission technique would interact with the same boundaries, but good bonds would have high transmission, while poor bonds would have decreased transmission.

Work by Gericke (80) in 1963 showed that UT techniques for metals and other solid materials were of limited value because they did not determine the geometry of concealed defects when these defects were comparable in size, or smaller than, the width of the UT search beam. Whenever such relatively small flaws were involved, UT pulse-echo testing at a single frequency yielded flaw location, but no information about its geometry. If a UT signal containing a wide band of frequencies is used (a white source), then the form and spectral energy distribution of the reflected beam are influenced by the geometry of the defect. Thus, an analysis of the defect echo yields information on the defect configuration. Frequency and time features can be used to characterize the defect that produces the ultrasonic reflection.

Work by Adler and Whaley (81) showed experimental dependence of spectral variations within a reflected broadband ultrasonic pulse on the size and orientation of the reflector. An analytical model was developed assuming that the interference of the waves received from the edges of the reflecting surface was responsible for the variations of the received frequency spectra. Their work was aimed at developing a method for determining the size of arbitrarily oriented flaws of crack-like geometry. When an object was placed in the path of a sound beam, the object produced waves which constructively interfered (added in phase) for frequencies given by

$$f_n = nv/(2d \sin a) \tag{4}$$

where n is an integer, d is the diameter of the circular object in the path of the ultrasound, v is the velocity of the sound in the medium, and a is the angle of the transducer relative to the object.

Tattersall (82) found that flaws are commonly detected by their ability to reflect a burst of ultrasound back to the piezoelectric transducer. Relying on the existence of a reflected signal, however, is not adequate to detect the difference between a weak and a good bond because both good and bad bonds can reflect ultrasound. He developed a theory that provides expressions for the coefficients of the reflected and transmitted amplitude and phase. The formula for the reflected amplitude from an adhesively bonded area is

$$A = \frac{Z_1 - Z_2 + iw(Z_1Z_2/K)}{Z_1 + Z_2 + iw(Z_1Z_2/K)} \tag{5}$$

where Z_1, Z_2, and K are the ultrasonic impedances of the adherent and adhesive, respectively, and K is a constant. By determining the amplitude, the quality of the bond can be derived from the Argand diagram show in Figure 2-4.

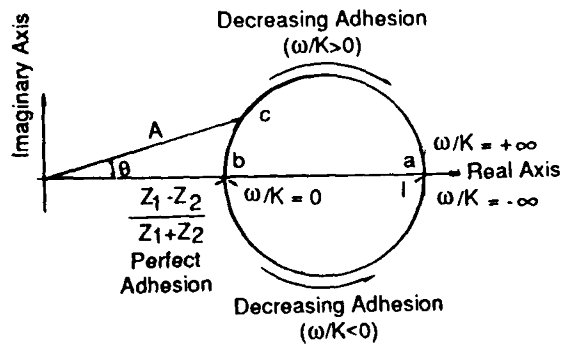

Figure 2-4. Reflected wave, A exp (iwt)K [Ref. (82), reproduced by permission of the Atomic Energy Research Establishment, Harwell]

Rose and Meyer (83) utilized ultrasonic immersion and spectroscopic procedures for predicting and evaluating bond quality in aluminum-to-aluminum step-lap joints made with Scotch-Weld 2216 structural adhesive joint. The UT tests were conducted using a medium-focused, 1/4-inch diameter, 10-MHz transducer in the immersion mode. The frontwall echo (FWE) and the backwall echo (BE) were compared. The results showed that as the ratio of FWE/BE increased, the adhesive bond quality increased. In other words, the adhesive was transmitting the ultrasonic energy through the adhesive when it was well bonded (see Figure 2-5). Therefore, the echo at the adhesive interface was decreased relative to the frontwall surface. Similar work has been conducted by Biggiero et al. (84).

Meyer and Rose (85) demonstrated the application and use of analytical models in the experimental ultrasonic evaluation of interface conditions in an aluminum-to-aluminum adhesively bonded system. Their results showed that a variation in bond quality due to surface preparation can be detected ultrasonically through careful inspection and signal-processing analysis. They worked with 50 step-lap test specimens. The bondline thickness was controlled to 0.01 inch. Half of the samples were cleaned in accordance with recommended procedures, and the other half were cleaned in the same way but without the chromic sulfuric acid etch. [The cleaning and bonding procedures were given in this paper (85).]

UT data were collected using 1/4-inch, 10- and 20-MHz transducers in an immersion mode. Several samples were destructively tested to determine the bond strength. Their results indicated that a variation in the interfacial condition of the bond was most noticeable ultrasonically by an amplitude change in the interface echo. As the quality of the interfacial bond decreased, the amplitude of the reflection from the adhesive-substrate interface increased.

The 10-MHz data did not correlate well; the 20-MHz data, however, showed clearly which sample had good surface preparation and which did not. Because some of the data were unclear, more experimentation is needed using the model developed by Rose and Meyer.

In 1979 Chernobelskaya, Kovnovich, and Harnik (86) developed a quantitative method of testing adhesive bonded joints using a high-resolution ultrasonic probe. The probe resolved echoes from the two interfaces of the bondline. The method was tested on aluminum-to-aluminum joints bonded with Metlbond 328 of Narmco Corp. Their method first tested the unbonded piece and obtained a set of echoes from the free surface, and then data were taken from the bonded joint. The effective reflection coefficient of the interface was derived from the measured amplitude ratio, which takes into account the loss in the aluminum. The true reflection coefficient can be derived from the measured transit times, thicknesses, and known densities of the aluminum and the adhesive according to $R = (Z2-Z1)/(Z2+Z1)$. The ratio of the effective reflection coefficient to the true reflection coefficient was the measure of the adhesive bond quality. Cohesive properties are also discussed as being related to the modulus of elasticity and ultrasonic absorption.

This model was simple; but it was only tested on two samples, and the data stated that the two samples were good. No

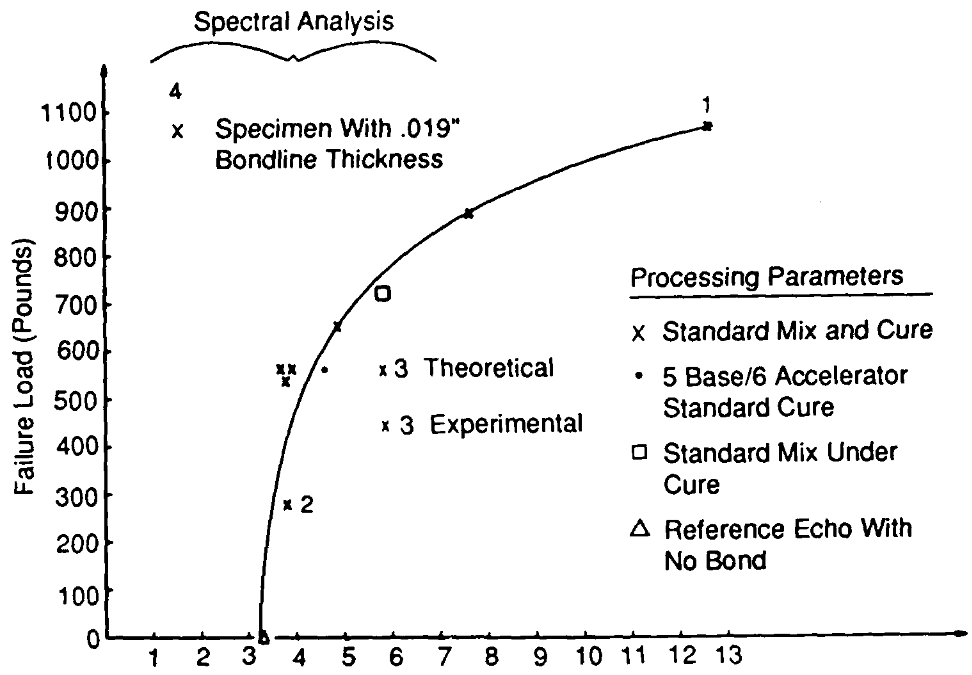

Figure 2-5. Curve showing failure load versus frontwall echo/backwall echo for an aluminum-to-aluminum step-lap joint with a 0.012-inch scotch-weld bondline thickness [Ref. (83), reproduced by permission of ASNT]

destructive tests were conducted on the samples to correlate the findings.

Alers, Flynn, and Buckley (87) felt that the basic premise on which ultrasonic methods can be used to predict the cohesive and adhesive quality of a metal-to-adhesive bond is based upon the argument that measurable changes in the elastic properties of the adhesive or the interface should be associated with changes in the cohesive or adhesive strength. Their work showed that the cohesive quality of a bond can be predicted from quantitative measurements of the velocity of sound and the attenuation in the adhesive layer.

What needed to be addressed was the lack of cohesive strength within the bulk of the adhesive and the lack of adhesive strength at the polymer-to-metal interface. When adhesive cures, it becomes hard and less attenuative to ultrasound. By measuring these two parameters, a cure-monitoring system can be made. Alers pointed out that the bond is really not between the adhesive and the metal, but between the oxide of the metal surface and the primer. The oxide/primer interface is usually between 0.02 and 0.2 micron thick, and NDE test has to target this thickness.

Expressions for the various reflection coefficients were given based upon impedances. For very thin adhesive layers, the resonance and antiresonance dips become coupled and it is difficult to determine the thickness and adhesive properties without a computer.

The cohesive strength of a polymeric adhesive should be closely correlated with its bulk physical properties. An improper mixture of chemical constituents of an incorrect curing procedure is apparent in cohesive strength and also modifies the ultrasonic velocity and attenuation. The Fourier transform of the adhesive interface reflection is

taken and divided by the Fourier transform of the front surface reflection. The minima in the spectrum is related to the layer thickness, and the depth is related to the attenuation as a function of frequency. The ultrasonic attenuation showed a "knee-type" curve relationship with the bond quality (as shown in Figures 2-6 and 2-7). Velocity was linearly related to bond quality. The resonance frequency was correlated with bond quality, but showed that the shift in resonance frequency was small for changes in adhesive strength.

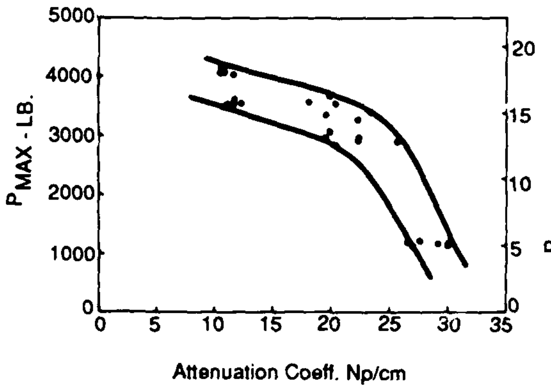

Figure 2-6. Correlation between the measured attenuation of the adhesive and the strength of the adhesive bond [Ref. (87), reproduced by permission of ASNT]

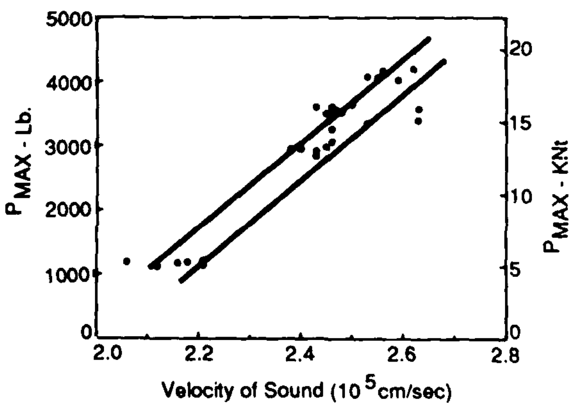

Figure 2-7. Correlation between the measured velocity of sound in the adhesive and the strength of the adhesive bond [Ref. (87), reproduced by permission of ASNT]

Segal, Thomas, and Rose (74) also used ultrasonic techniques for inspecting bonded structures. They found that the best ultrasonic technique for predicting adhesive quality was is high-frequency ultrasonics using signal processing, as described by Rose and Raisch (88). Williams and Zwicke (89) utilized various ultrasonic resonance methods to inspect adhesively bonded joints. They determined that neither simple nor more complicated ultrasonic approaches were able to absolutely assess joint quality.

A good bond is determined by several variables, and the ultrasonic interaction also depends upon variables that are difficult to separate. The authors categorized the bond variables into two areas: intrinsic and extrinsic. The intrinsic properties effecting cohesive bond strength included degree of cure, adhesive chemistry, bondline thickness, and voids. The extrinsic properties effecting the adhesive bond strength included surface cleanliness, surface contamination, etchant type and time, primer type and application, and wetting properties of the adhesive. They found that even if all the intrinsic properties were within proper tolerances, the bond may still fail due to extrinsic factors. They included joint geometry and loading, and the presence of defects.

A multidisciplinary approach was required that combined NDE and fracture mechanics to form a basis for a comprehensive quality assurance solution. NDE could detect defects and measure intrinsic cohesive and adhesive properties and, in the case where no defects were present, could assess bond quality. Fracture mechanics was employed to determine acceptable defect sizes (to be detected by NDE) for a given set of intrinsic bond properties. The interaction of ultrasonic waves with certain properties of the bond were placed in a table by the authors. (See Table 2-1) For example, longitudinal (L)-waves were strongly influenced by the cure state and bondline thickness, but did not do well for chemistry, porosity, etch, defects, or surface condition.

The experimental work was conducted on 6061 aluminum using Eastman EA 9649 adhesive. The L-waves were generated

Table 2-1

EFFECTS OF BOND VARIABLES ON ULTRASONIC MEASUREMENTS
(S = Strong Dependence; W = Weak Dependence) [Ref. (89), reproduced by permission of ASNT]

Ultrasonic Measurements	Bond Variables						
	Cure State	Chemistry	Porosity	Bondline Thickness	Etch	Surface Condition	Defects
Longitudinal Wave Velocity, V_L	S	W		S	W		
V_L vs. Wavelength			W				
Longitudinal Wave Hysteresis Atten., α_{HL}	S	S			W	W	S
Longitudinal Wave Scattering Atten., α_{SL}			S			S	S
α_{SL} vs. Wavelength			S				
Shear Wave Velocity, V_T	S	W		S	S		
V_T vs. Wavelength			W				
Shear Wave Hysteresis Atten., α_{HT}	S	S			S	W	S
Shear Wave Scattering Atten., α_{ST}			S			S	S
α_{ST} vs. Wavelength			S				

with a PVDF transducer which had high internal damping and broadband frequency response (1 to 35 MHz). Shear (S)-wave work was conducted with an S-wave transducer using a UTRC proprietary couplant. The signals from the front and back surfaces were digitized and processed. UTRC developed a pattern-recognition analysis algorithm called REcursive Structure IDentification (RESID) based upon a theory developed by A. G. Ivakhnenko (90). The algorithm involved five features (S-wave velocity, L-wave attenuation, S-wave attenuation, L-wave attenuation versus frequency, and S-wave attenuation versus frequency).

The RESID algorithm was applied to ultrasonic amplitude data. These amplitudes, shown in Figures 2-8 and 2-9, were the positive and negative RF amplitudes of the reflections from the adherent/adhesive interface and the positive and negative RF amplitudes of the adhesive/adherent interface. The features used in the algorithms are listed in Table 2-2.

Using the developed algorithms, experimentally predicted bond properties such as predicted bond strength, predicted shear strength, and predicted percent cure were correlated with destructively measured properties, shown in Figures 2-10, 2-11, and 2-12.

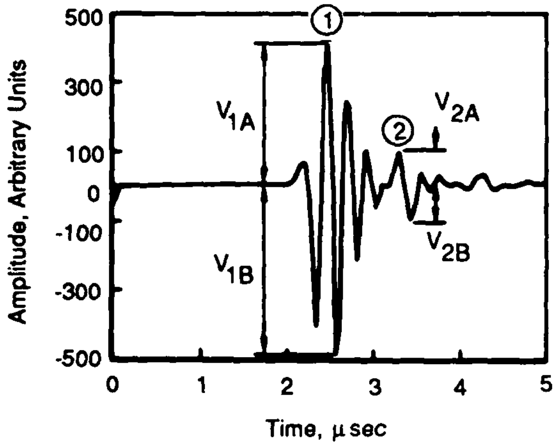

Figure 2-8. Typical time-domain waveform showing the reflections from the adherent/adhesive (1) and adhesive/adherend (2) interfaces [Ref. (89), reproduced by permission of ASNT]

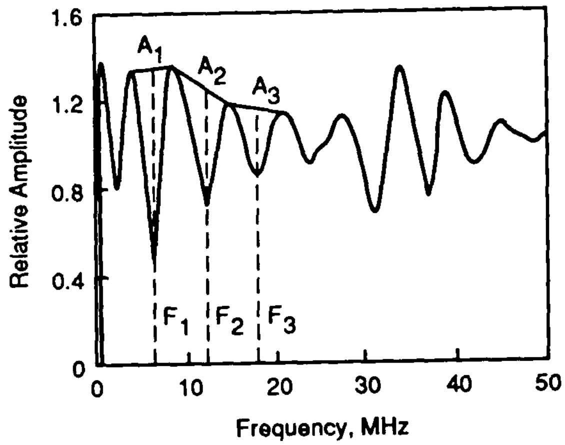

Figure 2-9. Typical deconvolved frequency-domain waveform using the adherent/adhesive interface reflection as the system response [Ref. (89), reproduced by permission of ASNT]

Table 2-2

ULTRASONIC FEATURES EXTRACTED FROM WAVEFORMS
[Ref. (89), reproduced by permission of the ASNT]

(a) Longitudinal Wave Features

Feature	*Determined by**	*Related to*
L_P	$(V_{2A} + V_{2B})/(V_{1A} + V_{1B})$	Total Attenuation, α_L
L_M	V_{2B}/V_{1B}	Total Attenuation, α_L
L_0	F_0	Longitudinal Velocity, V_L
L_1	F_1	V_L (Dispersion)
L_2	F_2	V_L (Dispersion)
L_5	$1/A_1$	Total Attenuation, α_L
L_6	$[\log(1/A_2)-\log(1/A_1)]/[\log F_1-\log F_0]$	$\alpha_L=\alpha_L(F)$
L_7	$[\log(1/A_3)-\log(1/A_2)]/[\log F_2-\log F_0]$	$\alpha_L=\alpha_L(F)$

(b) Shear Wave Features

Feature	*Determined by**	*Related to*
S_P	$(V_{2A} + V_{2B})/(V_{1A} + V_{1B})$	Total Attenuation, α_T
S_M	V_{2B}/V_{1B}	Total Attenuation, α_T
S_0	F_0	Longitudinal Velocity, V_T
S_1	F_1	V_T (Dispersion)
S_2	F_2	V_T (Dispersion)
S_5	$1/A_1$	Total Attenuation, α_T
S_6	$[\log(1/A_2)-\log(1/A_1)]/[\log F_1-\log F_0]$	$\alpha_S=\alpha_S(F)$
S_7	$[\log(1/A_3)-\log(1/A_2)]/[\log F_2-\log F_0]$	$\alpha_S=\alpha_S(F)$

*V_{1A}, V_{1B}, etc.; A_1, A_2, etc.; and F_1, F_2, etc. are defined in Figures 2-8 and 2-9.

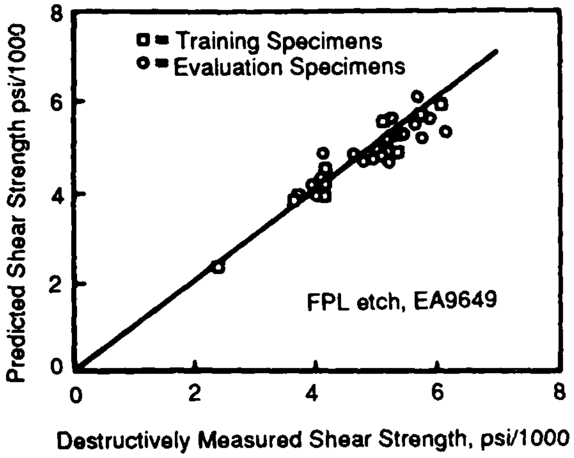

Figure 2-10. Experimentally predicted shear strength versus the actual shear strength in a defect-free specimen (measured destructively) as obtained using a RESID network [Ref. (89), reproduced by permission of ASNT]

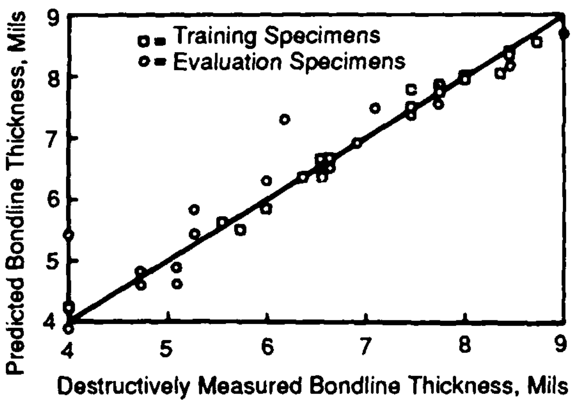

Figure 2-11. Experimentally predicted bondline thickness versus the actual bondline thickness (measured by sectioning) as obtained using a RESID network [Ref. (89), reproduced by permission of ASNT]

Rose, Avioli, and Bilgram (91) developed a combined NDE technique that made use of signal processing, pattern recognition, and a high-density ultrasonic scan into a hybrid-scheme for assessing the integrity of an adhesively bonded structure.

Experimentally, they used a 10-MHz transducer in the immersion mode and per-

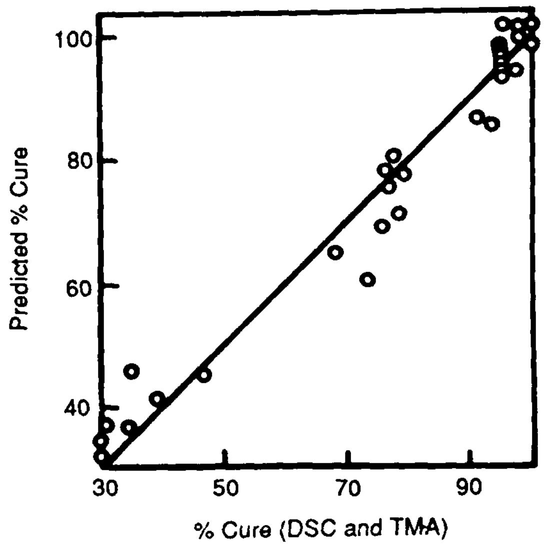

Figure 2-12. Experimentally predicted percent of cure versus the actual percent of cure (measured with differential scanning calorimetry, DSC, and thermometric analysis, TMA) as obtained using a RESID network [Ref. (89), reproduced by permission of ASNT]

formed a raster scan over the test plate. The test plate consisted of a sandwich configuration and contained curing defects, adhesive contamination, and voids in the adhesive. They looked at ten different samples. The RF waveform was digitized and analyzed on by several feature-extraction algorithms. The ratio of the adhesive layer reflection to the front-surface reflection was one of the major features.

2. *Spectral Domain Reflection/Transmission at the Bond Interface*

The spectral domain reflection/transmission techniques attempt to determine the quality of the bond. They do so by evaluating the interaction of the frequency content of the ultrasonic beam with the adhesive boundary interfaces. Frequency parameters such as center frequency, bandwidth, power spectrum, and others can be calculated from the Fourier transform of the reflected or transmitted ultrasonic beam. These features can be extracted from waveforms obtained from test samples that simulate various bond conditions

and an algorithm using weighted values of the features that are best correlated to the bond quality. If a good bond exists, then a certain algorithm can be developed. If a defective bond exists, then the algorithm gives a different value.

Chang et al. (92) analyzed the frequency spectrum of ultrasonic plane waves transmitted through a multilayered laminate structure at normal incidence to determine the amplitude distribution of the frequency components. In supporting the theoretical calculations, the wave equation was solved to evaluate the displacement field with the appropriate boundary conditions in a six-region laminate. Cavity resonance of the plane waves in the layers produced peaks in the transmission-frequency spectrum. Experiments were conducted using a pair of broadband acoustical transducers transmitting a pulsed ultrasound centered at 5 MHz through multilayer adhesively bonded aluminum plates with different thicknesses. Resonant peaks in the experimental frequency spectra were compared with those theoretically calculated from regions of good bond and debond.

The theory gave expressions for the transmitted energy and transmitted flux coefficient, and the effects of air gap and good adhesive layers were shown. Also, the effect of the adhesive-layer density, adhesive-layer sonic velocity, and adhesive sonic impedance on the theory were discussed. The experimental results looked promising, but more work was needed.

Chang, Couchman, Bell, and Gordon (93) in 1975 reported that NDE parameters evaluated from the ultrasonic spectroscopic method could be correlated with adhesive bond strength in multilayered structures. Selected portions of the reflected or transmitted RF signals were digitized; a Fourier transform was performed, and resonance peaks or antiresonance dips in the frequency spectrum characterized the thickness of the substrate plates and the adhesive gap. The shape and the Q-value of the peaks and dips determined the bond quality. The test samples were 2024 aluminum with PL717 adhesive. Each layer was 0.125 inch (0.317 cm) thick.

The ultrasonic pulse-echo ratio data were obtained using a 15-MHz transducer with a Panametrics 5052PR pulser/receiver. The Q of the resonance was related to the ratio (r) of acoustic impedances in the two media by

$$Q = 180/(\sin^{-1}(2r/r^2\text{-}1) \,. \qquad (6)$$

The Q values were measured at the half-width of the curves in the frequency spectrum.

The UT P-E amplitude ratio did not give good correlation, but did show a generalized trend of A1/A2 decreasing as bond strength increased (where A1/A2 is the ratio of signal amplitude for the top and bottom adhesive interfaces). The shear strength was found to vary directly with Q-value.

Chang, Yee, and Couchman (94) used an ultrasonic-frequency, spectral-analysis technique for detecting flaws in composite materials. This technique depended on the phenomenon of resonance interference of acoustical waves in materials. When the material thickness was an integral multiple of the half wavelength of the sound waves, destructive interference of a return echo by multiple reflections in the materials produced antiresonance dips in the frequency spectrum for the reflected signal.

Their experimental results showed that a well-bonded region of the composite had a very different frequency spectrum from a delaminated region. They could detect defects as small as 1.5 mm but were not able to differentiate among different sizes of defects. (See Figure 2-13.)

Chang et al. (95) utilized ultrasonic spectroscopy to evaluate the quality of the bond. They theorized that in adhesively bonded structures, destructive interference of the pulsed sound waves at the boundaries of the bond layer produced spectral information characteristic of the bond. The bondline thickness could be determined accurately from the frequency minima in the spectra. The width of the antiresonance dipped at half-power points, and the amplitude of the dips were related to the acoustic properties at the interfaces of the adhesive layer.

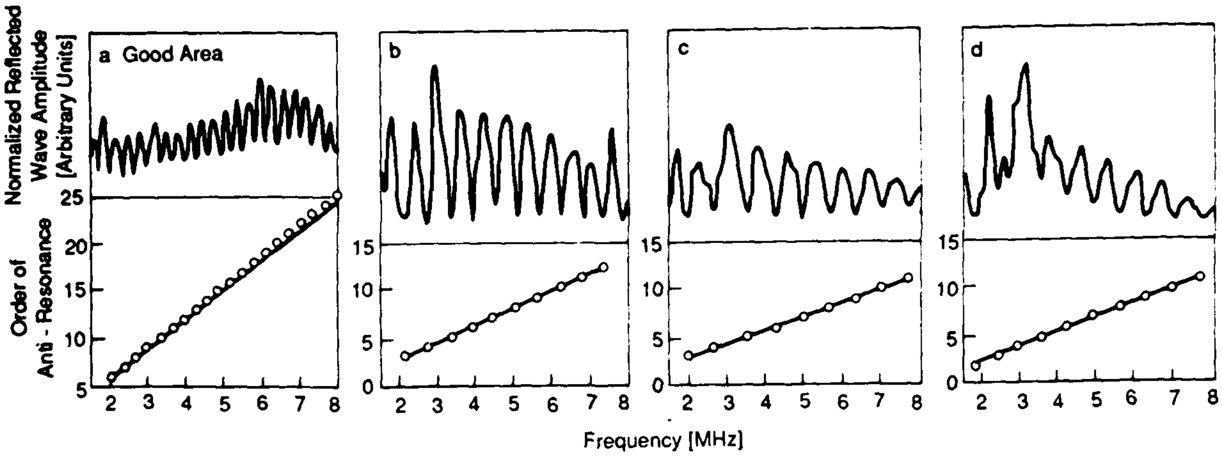

Figure 2-13. Plots showing how the spectra from flat-bottom holes (b: 6mm, c: 3mm, d: 1.5mm) differed considerably from the spectrum for the sound area (a). [Ref. (94), reproduced by permission of IPC Magazines Ltd.]

They provided an analytical calculation for the sound-wave interference in the bondline using a notation developed by Brekhovskikh (96). This calculation included the ultrasonic attenuation of the adhesive layer which, in effect, decreased the amplitude differences between the resonance peaks and dips in the frequency spectrum. Also, the acoustic impedance was directly related to the elastic modulus of the adhesive by $A = (E p)^{1/2}$ where E was the modulus and p was the density.

The experimental setup used 15-MHz, L-wave pulse echo; a wideband pulser/receiver; and plates of 2024 Al and RB-398 adhesives. Fabrication processes included low curing temperature, unetched substrate surfaces, adhesive sheet cutout, and insufficient bonding pressure.

Their test results showed that the amplitude ratio correlated well with bond quality. Also, excellent correlation between the reciprocal of the half-power bandwidth (1/B) and shear strength was found (see Figure 2-14). The amplitude ratio and the 1/B had a linear relationship, which provided a basis for a mathematical model to predict bond quality.

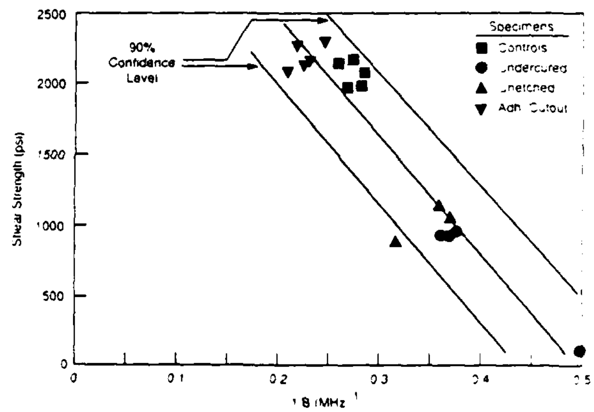

Figure 2-14. Correlation of ultimate shear strength with 1/B [Ref. (95), reproduced by permission of the Institute of Electrical and Electronics Engineers, Inc.,© 1976]

Raisch and Rose (88) examined the potential of selected ultrasonic signal features and the ability of these features to predict performance on an adhesively bonded structure. Adhesive, rather than cohesive, problems were considered. They claimed that the cohesive strength prediction had been solved by Flynn (97) using measurements of wave speed and attenuation in the bulk adhesive,

provided that adhesive-type failure did not occur. The cohesive-strength prediction problem was also studied by Yee, Chang, and Flynn (98) using ultrasonic spectroscopy measurements to characterize the physical state of the adhesive in a bonded laminate.

The experimental work of Raisch and Rose (88) was conducted using 0.25-inch (0.63-cm) diameter, 10-MHz transducers at normal incidence in the immersion mode. The data were acquired, digitized, and then operated in the amplitude-time and amplitude-frequency domain. Thirteen features were extracted for evaluation. Those in the amplitude-time domain included (1) peak-to-peak pressure variation, (2) the activity region (obtained by using a spline curve containing all the relative maximums of the rectified bond echo), (3) the area enclosed by the rectified amplitude-time bond echo within the activity region, and (4) the ratio of positive-to-negative area enclosed by the amplitude-time bond echo within the activity region. The remaining nine features were in the amplitude-frequency domain. They included (1) the maximum amplitude of the Fourier Spectrum, (2) the 6-dB down point on each side of the maximum amplitude frequency peak, and (3) the number of relative maximums and minimums occurring within the bandwidth region of the spectrum. The frequency locations of the maximums and minimums that occurred at lower and higher frequencies relative to the spectrum maximum also were picked, and then ratios of these amplitudes and their associated frequencies were used. Using this technique on 45 specimens yielded an overall 84-percent accuracy of calls.

Rose and Thomas (99) showed that ultrasonic C-scan techniques can be used to detect delaminated bonds. A completely automated ultrasonic inspection system was developed for predicting bond quality in metal-to-metal bonded step-lap joints. Results to date provide a 91-percent reliability for solving the problem of predicting the adhesive bond performance. The system used pattern-recognition techniques, especially the nearest neighbor rule and the Fisher linear discriminator algorithm, which separated the bond data into strong and weak bonds.

In the tests, a 10-MHz transducer was used. The results using three different transducers showed correct prediction of 95, 95, and 89 percent (the training set was done with the first transducer). However, the algorithm was still felt to be very much transducer dependent (especially on the pulse shape). A transducer test was developed to evaluate the use of different transducers. This deconvolution basically made all the transducers equal in their capability to predict good versus bad bond.

Ultrasonic pulse waveforms reflected from the interface of bonded joints were digitally recorded by Alers and Elsley (100). Certain frequency-dependent features of the reflected waves such as the lowest frequency of resonance and the highest split resonance frequency were correlated to the mechanical strength of the bond. The pulse-echo method was used in the immersion mode. The signals from the top of the metal surface and from the interface bonded region could be separated. A drawback of looking just at the amplitude ratios was that the bond strength was really determined by a very thin layer at the metal-to-adhesive interface, and the reflection from this critical layer was dominated by the metal-polymer impedance discontinuity rather than the structure of the interface. Their method was to Fourier transform the entire signal through the composite structure and to look at the frequency features.

The results showed that resonant frequency was very dependent upon bondline thickness. Other frequency-dependent variables needed to be evaluated and then correlated to the bond strength.

Thomas and Rose (101) studied the problem of predicting adhesive bond performance for both surface preparation and undercure defects using an ultrasonic experimental test bed system. The experimental test bed incorporated ultrasonic and computer equipment necessary to acquire and process data from various types of adhesively bonded test specimens. A set of 154 bond specimens was used to develop an algorithm that was 91-percent reliable in separating good and poor bond samples. A Fisher Linear Discriminant function was selected by the test bed as the

best pattern-recognition routine for this classification problem.

The features selected were:

- Peak-to-peak ratio of the echo and the reference signal-frequency shift
- Peak frequency
- Deepest depression frequency (dip frequency)
- Difference between the frequency of the dip and the peak frequency
- Ratio of dip amplitude to peak amplitude
- Difference between the first- and second-dip frequencies
- Standard deviation of the transfer function
- 6 dB beam width of the first dip.

E. A. Lloyd (102) worked with A-scan and frequency spectrum data from one substrate of the adhesively bonded composite. Using the same transducer and equipment, he took data through the adhesively bonded substrate and compared the two sets of data. The unbond condition was expected to be similar to the data taken from the substrate. A discussion of the theory was given, which basically said that when the transducer was over the bonded region, a mixed frequency spectrum was obtained, which was a measure of the compliance of the adhesive layer.

Recent work conducted by N. R. Joshi (103) also utilized the ultrasonic spectrum obtained from the interface region of the bond. The method he used was called the Chirp-Z Transform (CZT). The CZT is obtained by taking the fourier transform of the A-scan (time-amplitude data display) and determining the location of dips in the frequency spectrum. These dips are caused by interference between the bond interface layers. The frequencies of the dips were used as center frequencies in the CZT to expand the frequency resolution.

Joshi tested his technique on symmetric double-lap specimens fabricated from 6061T6 aluminum and the Dexter Hyson adhesive EA 9628. The samples had different surface preparations and were fabricated in different humidity level environments. His conclusion was that the frequency dip intervals or the successive cutoff frequencies could be correlated to the condition of the surface preparation and the level of humidity under which the bond was made.

3. *Low Frequency*

Thompson, Thompson, and Alers (77) reported results of several experiments that suggested the adhesive bond quality of honeycomb sandwich panels can be nondestructively determined by measurements of vibrational response. The data showed that both the damping characteristics of panel vibrations and the velocity of the propagation of elastic waves traveling along the surface and sample bondline can be correlated with destructively determined bond strengths. These results were a consequence of the viscoelastic properties of the adhesive, whereby changes in the elastic moduli and damping capacity of the adhesive can be related to the molecular structure and hence the strength of the adhesive.

Data collected on honeycomb at 7 kHz showed a relationship between the modulus and cohesive strength of the bond with the damping of the UT signal. The frequency and temperature at which the damping reached its maximum were interrelated through the expression $\omega t = 1$ where ω is $2\pi f$ and t is the relaxation time according to the Arrhenius equation

$$t = t_0 \, e^{W/kT} \qquad (7)$$

where t_0 is a temperature-independent factor, W is the activation energy, k is Boltsmann's constant, and T is the absolute temperature.

Damping of the vibrations of the adhesive rod specimens also correlated with cure and strength. They plotted the log decrement (decrease) of decay of free oscillations measured with bond strength. The data showed that the lower the adhesive bond, the higher the decrement (see Figure 2-2).

Averbukh and Gradinar (104) conducted work with the Fokker Bond Tester. They placed the piezoelectric transducers on a sheet of material and determined the acoustic characteristics. Then the composite material (composed of the first sheet cemented to another sheet) was monitored to determine its acoustic characteristics. The composite produced a shift in the frequency and acoustic impedance. The shift in frequency was indirectly proportional to the bond quality. The Bond Tester, however, had some shortcomings: (1) it required that the adhesive strength exceed or be equal to the cohesive strength and (2) it imposed sometimes unattainable technological requirements on the cementing.

Budenkov et al. (105) showed that the bond tester and the Stabmeter work only where the adhesive strength was higher than the cohesive strength. Bond quality can be determined by correlating the strength and the characteristic impedance of the glue using ultrasonics.

4. *Rayleigh and Surface Waves*

Staecker and Wang (106) studied the application of acoustic Rayleigh wave propagation in a layered medium. A theoretical derivation was made for the propagation in a fluid between two solids. Experimentally, they used 30-MHz Rayleigh waves of 2-microsecond duration on the exposed region of the second layer (see Figure 2-15). The wave traveled into the bonded region and excited characteristic waves of the layer. Their work demonstrated that acoustic waves could be propagated in a solid-fluid-solid guiding structure.

Thompson et al. (77) also did some surface work and found that the surface-wave velocity increases with the adhesive bond quality.

5. *Lamb and Interface Waves*

Rokhlin (107) analyzed diffraction of Lamb waves by a finite crack situated on the plane of symmetry of an elastic layer. The surface of the crack and the layer were assumed to be stress free. The field of the reflected and transmitted waves as well as the field in the vicinity of the crack were given as expansions of natural waves of the elastic layer. The amplitudes of these waves were found from exponentially converging infinite systems of equations.

The interaction of waves at bonded interfaces was studied by Jones and Whittier (108). They looked at plane-strain elastic-wave propagation for two dissimilar half-spaces joined together at a plane interface by an elastic bond. The bond thickness was assumed to be small compared to the wavelength; the existence of interface waves was shown to be governed by a parameter involving bond stiffness and wavelength. The infinitely stiff bond case (fully cured) would reduce to the Stoneley wave data; and the infinitely soft bond would produce two Rayleigh surface waves, one in each medium.

They also found that as the bond became stiff, the Rayleigh waves could change

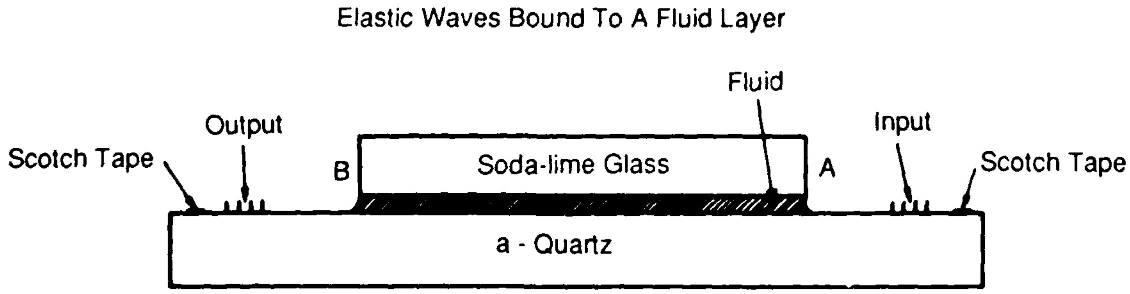

Figure 2-15. Experimental layered geometry configuration used to generate Rayleigh-wave data [Ref. (106), reproduced by permission of the American Institute of Physics]

into two interface waves, each involving motion of both media. One of these waves, the one with the higher speed, disappeared for a sufficiently stiff bond. With further increase in bond stiffness, the remaining interface wave either went into a Stoneley wave or disappeared, depending on whether or not Stoneley waves were possible. Furthermore, when the Rayleigh wave speed in the faster medium was greater than the S-wave speed in the slower medium, the Rayleigh wave in the faster medium existed only for zero bond stiffness. As soon as the stiffness became finite, the wave disappeared, and only a wave similar to the Rayleigh wave in the slower medium may occur.

A new technique for analyzing interfacial conditions in completed adhesive bonds was suggested by Claus and Kline (109). The method was based on the sensitivity of Stoneley waves, which propagated along the boundary between dissimilar solid media, to changes in the material properties of the interface region. Stoneley wave attenuation measured after polishing was found to increase as a function of increasing surface roughness in specimens of borosilicate crown glass bonded with an anaerobic cement to a substrate of 7740 Pyrex mirror glass.

While many NDE techniques have been used to detect large-scale defects such as voids or delaminations, no existing single NDE technique can detect "weak bond" condition. This work, therefore, was aimed at developing the Stoneley wave-measurement technique as a tool to determine the bond quality. The experimental setup utilized surface acoustic waves (SAW) from an x-cut piezoelectric crystal mounted on a conventional Rayleigh-angle water-coupled wedge. As the waves passed into the adhesively bonded region, they became Stoneley waves, which are affected by the boundary condition. The outgoing waves were detected with an optical interferometer. In these experiments, the Stoneley wave attenuation increased as the surface roughness of the two adherents increased.

The use of ultrasonic wave-velocity measurements of surface, plate, or interface waves for the evaluation of adhesive bond quality was studied by Pilarski (110). His work dealt with two classes of problems: (1) cases where a step-like change occurred in the acoustic impedance such as bimetals or glued metal bonds and (2) cases where the bond was a thin intermediate layer whose thickness was much less than the wavelength of the ultrasonic wave. The second case was when the layer was approximately equal to the wavelength of the ultrasonic waves.

One way to evaluate the adhesive quality of a layered joint is based upon the measurements of the pressure coefficient of the reflected wave for the bond interface. Another way is to use ultrasonic waves propagating parallel to the bond surface. These waves are called subsurface waves in the case of a layer on a base, plate waves in the case of one or more solid layers, and interface waves in the case of two elastic half-spaces (i.e., a solid medium of thickness several times the wavelength of a surface mode). Previous work had used the decay of the surface, plate, or interface waves in evaluating the bond quality.

The purpose of Pilanski's paper (110) was to use velocity measurement of waves propagating along the bonded surfaces to determine the bond quality. The theory considered two types of boundary conditions, i.e., welded and adhesively bonded boundaries. For the welded case, a continuity of displacement and stress occurred. For the adhesively bonded case, a vanishing tangent stress can occur. Decrease in adhesive quality yields a decrease in the phase velocity of the surface waves, as shown in Figure 2-16.

Experimentally, the phase velocity of the ultrasonic waves can be measured using critical-angle reflectivity. The change in the critical angle is related to the change in velocity which, in turn, is related to the quality of the bond. In general, the higher the phase velocity, the greater the bond.

Most of the previous UT methods used for determination of quality of adhesive bonds used 0-degree L-waves, which are not sensitive to the adhesion properties between the adhesive and the adherents. Rokhlin (111) used guided waves to produce shear

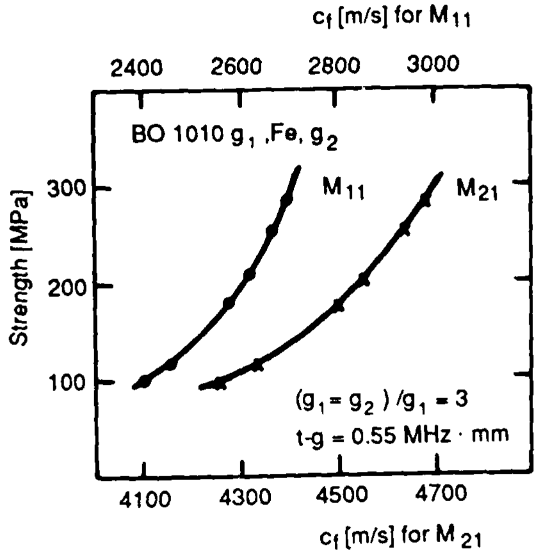

Figure 2-16. Relationship between phase velocity of the first two modes of plate waves and shear strength of the adhesive joint in bimetal [Ref. (110), reproduced by permission of ASNT]

stress on the interface. Interface waves were applied for monitoring curing of the adhesive, and Lamb waves were used to study adhesive joints of the thin sheets.

An interface wave can be obtained in the adhesive joint by generating a surface wave into the lower substrate. The thickness of the layer must be smaller than the UT wavelength. Bond failure can occur inside the adhesive (called cohesive failure) and between the adhesive and adherent (called adhesive failure). Using the interface waves, the following can be observed:

- When contact between the adhesive and the substrate is not ideal, the interface wave velocity decreases.
- By monitoring two interface waves, one through an unbonded region (calibration) and one through the region that is adhesively curing, as the curing of the adhesive cures, the phase velocity between the two sets increases. (See Figures 2-17 and 2-18.)

When the thicknesses of the bonded substrates are comparable to the wavelength, Lamb waves are used. The dispersion characteristic of Lamb waves can be useful to monitor the slip (liquid) and rigid (bonded) boundary conditions. During the curing process (liquid to solid), the Lamb wave propagation mode is transformed with a change in phase from the mode in the slip condition to the mode in the rigid condition.

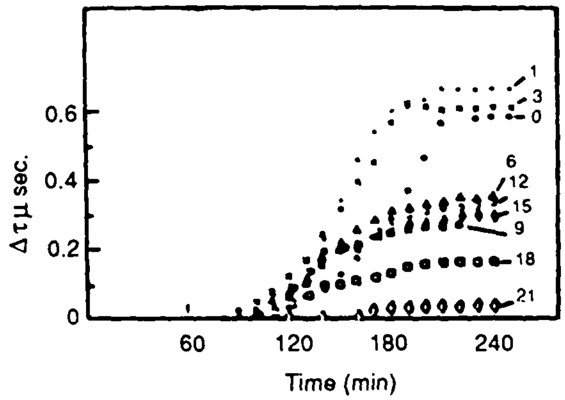

Figure 2-17. Change in the interface wave velocity as a function of curing time for aged specimens [Ref. (111), reproduced by permission of the National Aeronautics and Space Administration (NASA)-Lewis]

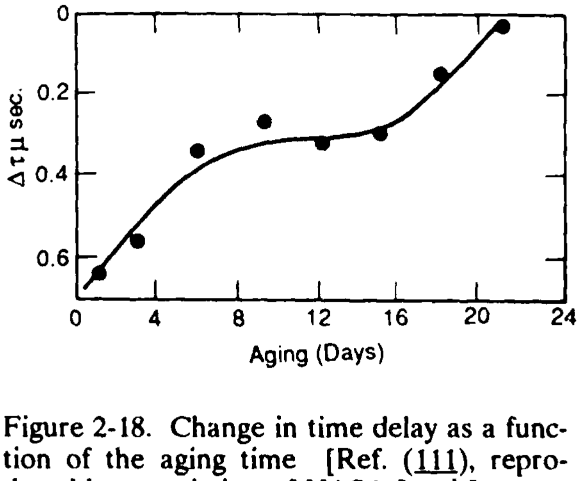

Figure 2-18. Change in time delay as a function of the aging time [Ref. (111), reproduced by permission of NASA-Lewis]

Data were collected using 0.5-, 1-, 1.5-, and 2-MHz transducers on adhesive films that were 80 to 90 microns thick (approximately 3 to 4 mils). The 1- to 2-MHz data were good; the 0.5-MHz data, however, did not show good results due to edge problems.

Rokhlin, Hefets, and Rosen (112) studied a thin adhesive film between two bonded adherents and showed that it was capable of localizing the energy of elastic waves in the form of an interface wave. The phase velocity and transmission losses of the interface wave were measured during the course of polymerization of the adhesive. The phase velocity of the interface wave and the effective shear modulus of the interface film were related to the quality of the adhesive bond, as shown in Figure 2-19.

The thin film located between plates exhibited waveguide properties if the shear modulus of the film were smaller than that of the substrates. When the film thickness was much smaller than the wavelength, the interface wave produced shear stress only in the film. This worked in the following way. First, a Rayleigh wave was propagated on the surface of the top part. If two plates were put together with a thin layer between them with the layer thickness much smaller than the Rayleigh wavelength and much larger than the hydrodynamic boundary layer, then the phase velocity of the interface wave (V_l) was smaller than the Rayleigh wave velocity (V_r). The relationship is

$$V_l/V_r = 1 - KthBr\rho_0/4pa_r \qquad (9)$$

where $B_r = (a_r^2-1)^{1/2}$, and $a_r = K_r/K_t$ are the wave number for the Rayleigh and shear waves in the substrates, ρ is the substrate density, and h is half of the layer thickness.

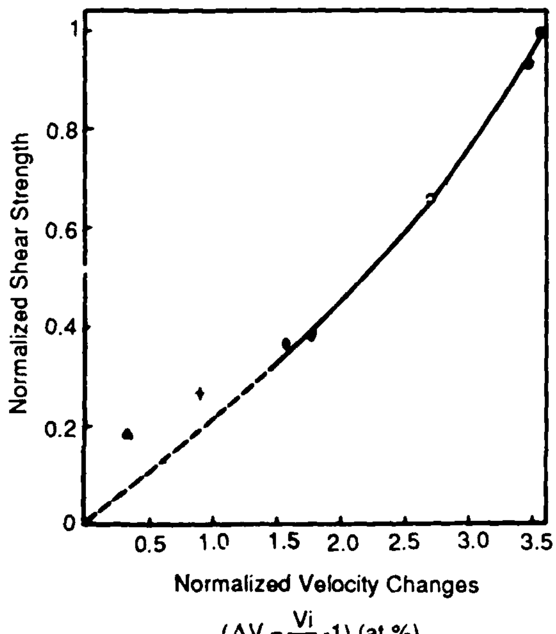

Figure 2-19. Experimental relationship between the interface wave velocity and the normalized shear strength [Ref. (112), reproduced by permission of the American Institute of Physics]

Now if the layer thickness were smaller than the boundary layer, then the shear displacement was not damped out across the film thickness and was transmitted from the lower to the upper substrate without a break in continuity. Thus, as the film solidified, the phase velocity tended toward the shear velocity. The measured velocity and damping factor of the interface waves could be used to calculate the complex shear modulus of the interface film. When there was an absence of shear bonding between the adhesive and at least one substrate, then velocity of the interface wave was close to the Rayleigh wave velocity.

Two 1.2-MHz narrow-band transducers were used for the tests. The greater the change in velocity, the greater was the quality. Also, the greater the transmission loss, the greater was the quality.

Rokhlin, Hefets, and Rosen (113) also studied the waveguide properties of a thin film separating two elastic half-spaces possessing a shear modulus higher than the shear modulus of the film. It was shown analytically and experimentally that such a film was capable of localizing the energy of elastic waves near the interface. The velocity of the interface wave was determined by the shear modulus, density, thickness of the film, and elastic properties of the substrates. The velocity of the interface wave was between the

Rayleigh and the shear wave in the solid substrates. It was shown that the interface wave could be used to estimate the elastic and dissipation properties of thin interface layers.

6. *Stress-Wave Factor*

Vary et al. (114-116) showed that the ultrasonic parameter called the stress-wave factor correlated with the tensile strength and interlaminar shear strength of graphite fiber composites. Williams and Lampert (117) looked at impact damage to composites and compared the through-thickness attenuation and the stress-wave factor measurements to the degradation.

Experimentally they used two 0.1 to 3 MHz, broadband through-transmission transducers to determine the attenuation through the damage composite, and a 0.1 to 3 MHz, broadband-transmitting transducer with a 375-kHz receiving transducer to measure the stress-wave factor. The stress-wave factor was defined as

$$e = g\ r\ n \tag{8}$$

where g is the accumulation time after which the counter was automatically reset, r was the transmitter repetition rate, and n was the number of cycle oscillations exceeding a fixed threshold in the output waveform generated by the input pulses. The stress-wave parameter characterized the specimen length, while the through transmission characterized the specimen's thickness. They concluded that both inspection techniques provided adequate means to determine quality of the composite.

Dos Reis, Bergman, and Bucksbee (118) conducted tests to evaluate the adhesive bond quality between rubber and steel plates using the stress-wave factor measurement technique. This technique measured the relative efficiency of energy transmission into the specimen. A UT pulse was injected with a transducer mounted on the surface of the specimen. The number of oscillations higher than a chosen threshold were monitored. The more internal damage, the greater was the attenuation and the less efficient the energy transmission (lower ringing). The ringing was detected by another transducer on the surface.

Experimentally, the stress-wave factor measurements were obtained using the portable acoustic emission technology (AET) AU instrument. The pulsing transducer had a flat response from 0.1 to 3 MHz. The receiving transducer was resonant at 375 kHz. The injected pulse was 150V at a rate of 250 pulses/second.

7. *Horizontally Polarized Shear Waves*

Horizontally polarized shear (SH) waves are generated with special transducers that emit shear waves at 0 degrees. In order to propagate SH waves, a viscous couplant must be applied to effectively couple the shear waves into the part.

Yew (119) performed a preliminary study using SH waves on a bonded plate to estimate the bonding quality of the adhesive bond. The concept was that the characteristics of the SH waves in the bonded structure were dependent upon the bonding stiffness of the adhesive; thus, an estimation of the adhering strength can be made by observing the behavior change of the SH wave motion. Two measurable modes of SH waves are in the structure during the early stages of adhesive curing, and the amplitude of the second-mode SH wave decreases as the adhesive cures and finally disappears after the adhesive-curing process is completed.

In addition, Yew studied the propagation of Stoneley waves in the interface of the two bonded solids. It was shown that the attenuation and the propagation speed of sonic waves in the medium were related to the bonding strength and the stiffness of the adhesive. A mathematical model for using SH waves to determine bond quality was described. When the cohesive strength was low, the wave behavior of the bond was similar to that of a free plate. The wavelength of the applied SH wave was so long in comparison with the plate thickness that a standing wave was established along the thickness of the plate.

The experimental tests were conducted on aluminum test plates bonded with DER 332 epoxy resin with a T403 amine curing agent and 399 accelerator. The SH

were generated with a 0.5-MHz transducer. A 600-kHz wave-train frequency, 30 microseconds long, was delivered to the strip at a rep rate of 500 Hz using an Arenburg pulsed oscillator. After the wave train had traveled 4 inches (10 cm), it was detected by a 5-MHz, SH transducer. This set up two modes in the plate. The second mode signal clearly decreased as the adhesive cured, while the first mode stayed relatively constant. One problem with this technique was ensuring proper coupling of the SH waves. Another issue was that the width of the receiver transducer should be shorter than the SH wavelength.

8. *Obliquely Incident Ultrasonic Waves*

Obliquely incident waves refer to ultrasonic energy introduced into a part at large angles relative to the normal of the part (angles usually above 60 degrees from the normal). In many cases, the reflection coefficient measured at the bond interface peaks at a relatively high or obliquely incident angle.

Using obliquely incident waves, Rokhlin and Marom (120) demonstrated that amplitude changes of the reflected waves during adhesive curing occurred simultaneously with changes of the wave velocity in the adhesive. The sensitivity of the method to evaluate the interface properties was higher for thinner adhesive-interface films. It was shown by Kuhn and Lutsch (121) in 1961 that the reflection coefficient for obliquely incident L-waves from a slip interface may be higher in some cases than from a free surface; thus, the value of the reflection coefficient can be used for evaluating the interface properties.

Bulk L-waves are insensitive to the existence of a thin liquid layer at the interface, which exhibits no shear strength resistance. To measure properties of the adhesion between the adhesive and the substrate and to monitor the adhesive cure, shear deformation of the interface must be induced. The theory was discussed, and optimum angles for inspection were derived.

The experiment was discussed. They measured (1) the amplitude of the reflected signal at the joint and (2) the L-wave velocity and attenuation in the bulk of the sample. The change in phase velocity was determined by measuring the phase shift of the RF signal. Transducers used were 1.3 MHz. The reflected signal amplitudes as a function of cure time for three different adhesive thicknesses (8, 35, and 75 microns) were plotted, as shown in Figure 2-20. The thinnest showed the most dramatic effect, changing approximately 11 dB over the cure cycle, while the thickest samples only changed 3 dB over the cure cycle. That is, the technique worked best when the ratio of the adhesive layer thickness to the UT wave velocity was approximately 0.01.

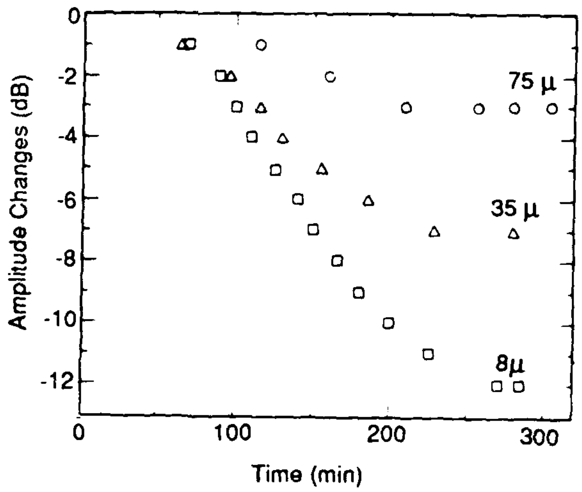

Figure 2-20. Dependence of the amplitude of the reflected signal on the time of adhesive curing for different thicknesses of the adhesive (metal-metal bond) [Ref. (120), reproduced by permission of the American Institute of Physics]

Similar results were obtained by Pilarski and Rose (122) using obliquely incident shear waves. They looked at adhesively bonded aluminum samples and found that the reflection coefficient as a function of angle through a bonded structure varied as a function of the bond rigidity.

D. Acoustic Emission Techniques

Acoustic emission (AE) techniques utilize piezoelectric transducers primarily to receive

high-frequency pulses generated in a material by some change within the material such as defect initiation or growth.

Acoustic emission (105) can be used to evaluate bond quality. A defective bond produces a large increase in AE during loading. This method, however, is not being used in practical applications because it has not been sufficiently developed or have been applied for only a few cases. Averbukh and Gradinar (104) collected data on bonded composites and found that the amount of AE as a function of load varied depending upon the quality of the bond.

E. Holographic Techniques

Holography is a fairly complex optical technique which requires a laser, a test object, and an identical reference object. The laser light illuminates both the reference object and object under test. Any differences in the reflected light from the object under test compared to the reference shows up as a change in the diffraction pattern. Changes on the order of a fraction of a wavelength of the laser light can be perceived (on the order of microinches).

Mueller, Gupta, and Keating (123) developed a holographic weak-signal-enhancement technique (WSET) which enhanced the image of the source of a weak signal of interest in the presence of an unwanted strong signal with very little *a priori* knowledge. The feasibility of the WSET was shown by simulating a two-solid-layer acoustic problem on a digital computer. The enhancement method was based upon the use of the signal field for reconstruction and high-pass filtering of some of the information.

F. Radiographic Techniques

Radiographic techniques refer to the use of penetrating atomic or nuclear particles to image an object. The most common ways to obtain penetrating radiation are by using an isotopic gamma-ray source, x-ray tube, or neutron source. Each provides radiation to illuminate an object. The radiation passes through the object under test and is usually imaged on film. Electro-optical devices also can be used to receive the penetrating radiation and generate an image.

Gamma-ray or x-ray penetration is affected by the condition of the material such as density variations and the presence of voids or inclusions. This type of radiography can be used to detect damage to honeycomb, presence of debris and contaminants, and lack of bond due to absence of adhesive (72).

Neutrons are affected more by nuclear processes than density variations such as proton-neutron scattering. Most organic adhesives contain between 8-and 12-percent hydrogen (proton), so neutron radiography becomes a viable tool to inspect for adhesive problems. Dance and Petersen (124), using a californium-252 source, demonstrated that neutron radiography could be used to detect defects in adhesives.

G. Thermography

Thermographic techniques utilize heat energy and monitor how this energy interacts with and travels through the object under inspection. The wavelength of this energy is usually in the range of 8 to 20 microns. In order to detect or image this energy, special detectors are needed.

Thermography can be separated into two methods, passive and active (75). The passive method utilizes an external heat source while the active method utilizes internal temperature changes when an external force is applied that produces internal frictional heating. The thermographic method makes use of the difference in thermal conductivity of a good bond and a poor bond. In the poor bond, there could be a discontinuity at the adhesive/adherend interface which would produce a poorer thermal conductor.

In practice, passive thermography utilizes an external heat source and an infrared camera (or other imaging system sensitive to thermal radiation). Monitoring of thermal transients is the most effective method. Monitoring of absolute temperatures is difficult because of variations in thermal emissivities, nonuniform heating, and many other problems. The defects appear as cooler regions (if

a through-transmission technique is used) because of the lower thermal conductivity through them. If the camera and the heat source are on the same side, then the defects will appear hotter because they do not transmit the heat as well. In addition, the sensitivity of the method decreases as the thermal conductivity and depth of the defect increase.

Work by McLaughlin et al. (125) and Reynolds and Wells (126) has shown that thermography can be used to detect delaminations and voids. Very little work has been done to determine its capability to actually determine the quality of the bond.

Active thermography makes use of an externally applied force and an infrared camera. For example, by vibrating the surface of a bonded structure, the defective regions will experience a local rise in temperature due to frictional heating. These defects can then be observed using the infrared camera. Work by Russell and Henneke (127) has shown that defects in composite structures can be detected if the vibrational frequency is that of the defect membrane resonance. Again, however, very little work has been conducted to determine the capability of active thermography to determine the quality of the bond.

Recently, a large amount of work has been conducted using thermographic techniques to detect the presence of debond. However, this work was presented as papers at seminars and technical meetings and has not yet been published.

H. Nuclear Magnetic Resonance

Nuclear magnetic resonance (NMR) is primarily used to detect the presence of hydrogen, especially in the form of moisture. This method has not been used to determine bond quality; however, it is believed that work should be conducted using NMR since one of the major causes of bond failure is the ingestion of moisture. This method requires the use of a large magnetic field and an RF exciter coil. This method cannot be used for metals, but should be of practical use for composite structures.

III. QUALITY CONTROL AND INSPECTION NEEDS

A. Materials

1. *Adhesives*

Quality control of the adhesives selected for bonding is very important to obtain bonded joints of desired strength (1, 4). Testing and inspection are needed to assure that the physical and chemical properties of the adhesives meet the required specifications. Properties requiring controls include mix proportion of the base and additive materials, fiber-resin volume ratio, mix homogeneity, gelation time, resin content, fiber alignment, moisture content, viscosity, flow, and tack.

2. *Adherends*

The quality of incoming adherend details comprising the joint must be inspected and tested to assure their meeting acceptance requirements (1, 4). Parameters such as size, thickness, material type, and general appearance as to distortion, corrosion, or defects require checking. Careful control of the subsequent surface preparation of the adherends is of paramount importance for production of reliable bonded joints (1, 4, 39). All the processes involved in the surface preparation--cleaning, rinsing, chemical treatment, drying, and/or priming--must be monitored and controlled. Physical and chemical properties of the materials used in these processes such as primer, solvent, water, and solution for chemical treatment must be tested and inspected for temperature, composition, and contaminants.

After surface treatment, the adherend needs to be checked for the wettability, surface roughness, uniformity of the treatment, and lack of contaminated area. If priming is involved, primer thickness, curing state, and presence of defects require inspection. In addition, temperature, humidity, and cleanliness of the preparation area must be monitored and controlled.

B. Bonding and Curing

Control of the bonding and curing processes is another key for a successful bonding (1, 4). During adhesive application, the thickness and uniformity of the adhesive must be monitored and controlled. For film-type adhesive applications, the presence of wrinkles, trapped air bubbles, or foreign substance such as release paper must be checked. For honeycomb sandwich structures, measuring and controlling the size of the fillet is required. Proper assembly and fitting of components or proper stacking sequence during lay-up must be monitored; and curing temperature, pressure, and time has to be measured and controlled. To control the curing process, the degree of curing must be monitored. After the adhesive is cured, the bonded area needs to be inspected for defects such as debonds and voids.

C. Inservice Inspection

The strength of the bonded joints and components degrades with service through development of environmentally induced defects such as cracks, debonds, delaminations, and corrosion. To ensure the structural integrity and safety of the bonded assembly, periodic inspections are essential.

IV. SUMMARY

This report has presented a large amount of information on the subject of adhesive bonding. Applications of adhesive bonding in use today were cited along with associated advantages and disadvantages. A generalized discussion of the bonding process and the parameters causing bonding problems were discussed as well as failure mechanisms. Finally, a brief but thorough coverage of the nondestructive methods utilized in the past to evaluate the condition of the bond were addressed.

Most of the information in the report deals with the use of NDE methods employed to date, involving such ultrasonic techniques as:

- Through-transmission or pulse-echo reflection of longitudinal waves with the adherend/adhesive/adherend layer (in both time and frequency domain),
- Stoneley waves interacting with the adhesive layer,
- Rayleigh waves,
- Obliquely incident waves, and
- Horizontally polarized shear waves.

Work using all these techniques has indicated results that correlate with bond quality in a limited number of tests.

Additional work has been conducted using sonic waves, and the body of evidence shows that sonic waves can be used to detect debonds. The "tap" technique has been used throughout the industry as a quick and easy method to detect large, near-surface delaminations.

Other methods also were evaluated including acoustic emission, optical holography, radiography, and thermography. Application of these methods to certain bonding problems has shown good success in differentiating between a bond and debond, but very little capability in quantifying the quality of the bond. Radiography in reality is used to detect defects such as inclusions and voids in the adherend/adhesive layer that can cause poor bond quality. Nuclear magnetic resonance has some potential for determining bond quality in nonmetallic structures by detecting the presence of moisture in the bond. Little work, however, has been done with this technique.

While many NDE techniques have been applied to assess bond quality, most of these can only determine whether there is a bond (of some unknown quality) or a complete lack of bond (debond). This differentiation capability of an NDE technique is indicated by an X in the bond/debond column in Table 4-1 on the following page. Other techniques have shown some promise in being able to determine if a bond is good, average, poor, or unacceptable. These categories are called bond quality, and the techniques with this capability or potential to distinguish bond quality are listed with an X under the bond quality column. Some techniques also can detect the qualities of the adhesive such as adhesive thickness or the presence of inclusions or porosity in the adhesive layer. These techniques are listed with an X in the inclusions and thickness columns.

Table 4-1

SUMMARY OF NDE METHODS AND TECHNIQUES APPLIED TO DETERMINE ADHESIVE BOND QUALITY AND AN EVALUATION OF THEIR CAPABILITIES

Method/Technique	Bond/ Debond	Bond Quality	Inclusions	Thickness
Sonics				
Coin Tap	X		X	
Mechanical Impedance	X		X	
Mechanical Resonance	X		X	X
Ultrasonics				
Pulse-echo	X		X	X
Through-Transmission	X		X	
Frequency Spectrum	X	?X*	X	X
Rayleigh Waves	X	?X*	X	X
Interface Waves	X	X	X	
Stress Wave Factor	X	?X*	X	X
Horizontally Polarized Shear Waves	X	X	X	X
Obliquely Incident Waves	X	X	X	
Acoustic Emission				
Passive	X		X	
Active	X		X	
Holography	X	?X*	X	
Radiography			X	X
Thermography	X		X	
Nuclear Magnetic Resonance	X	X		

*Under Bond Quality, ? denotes that these techniques have demonstrated some theoretical or experimental potential for determining bond quality.

V. CONCLUSIONS

Almost every NDE method has been utilized at one time or another in an attempt to develop a technique to adequately describe bond quality in a bonded structure (128, 129). Most of the work to date has centered around application of ultrasonic techniques. The interaction of the through-transmitted (or reflected) longitudinal (L) wave with the adherend/adhesive/adherend interfaces can be used to easily detect the difference between the good bond and the total debond. The L-wave, however, does not perturb the adhesive layer in a way that would provide a means to interrogate the strength of the adhesive layer or the adherend/adhesive interface.

Other ultrasonic waves such as the Stoneley and interface waves directly interact with the adhesive layer so that the strength of the adhesive or adherend/adhesive interface can potentially be measured. Unfortunately, the application requirements for these techniques make it almost impossible for field inspections. Horizontally polarized shear waves launched from the top surface into the material interact with the adherend/adhesive interface, but more work must be done to further evaluate this technique.

Based upon the amount of work conducted to date, the most promising approach for the largest variety of bonding inspections is application of one of the ultrasonic techniques. The information in this report is based upon multiple case studies, some of which were correlated with destructive testing; the one point, however, that seems apparent is that no one technique was tested on a large variety of different bond types with known bond strength.

A program should be conducted using a large number of ultrasonic techniques on a carefully produced set of samples that would cover most bonding situations. The test plan should be coordinated with both NDE as well as adhesive bonding experts. After a thorough evaluation, the samples should be destructively tested to determine the actual bond strength.

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